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Information Division**

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I. INTRODUCTION

NASA TECHNICAL MEMORANDUM

I. INTRODUCTION

The Microgravity Science and Applications (MSA) Program is directed toward research in the science and technology of processing materials under conditions of low gravity to provide a detailed examination of the constraints imposed by gravitational forces on Earth. The program is expected to lead, ultimately, to the development of new materials and processes in commercial applications adding to this nation's technological base. The research studies emphasize the selected materials and processes that will best elucidate the limitations due to gravity and demonstrate the enhanced sensitivity of control of processes that may be provided by the weightless environment of space. Primary effort will be devoted to a comprehensive study of the specific areas of research which revealed potential value in the initial investigations of the previous decades. Examples of previous process research include growth of crystals and directional solidification of metals in the quiescent conditions in which gravitational fluid flow is eliminated; containerless processing of reactive materials to eliminate reactions with the container and to provide geometrical control of the product; synthesis and separation of biological materials in weightlessness to reduce heat and mass transfer problems associated with sedimentation and buoyancy effects; identification of high vacuum characterization associated with an orbiting wake shield; and minimal knowledge of terrestrial processing methods.

Additional effort will be devoted to identifying the special requirements which drive the design of hardware to reduce the risk in future developments. Examples of current hardware studies are acoustic, electromagnetic, and electro-static containerless processing modules and electrophoresis separation devices.

The current emphasis on fundamental processing science and technology in selected areas will continue as the Microgravity Science and Applications Program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment.

Emphasis will be placed on the expansion of currently funded activities for ground-based and space flight investigations to maximize the outputs from these opportunities. Initiatives requiring new hardware will be encouraged at a low level until funds can be made available. The expansion of current efforts is occurring as a result of focusing support for current space flight investigations on forming facility experiment teams to provide advice and identify future involvement. Emphasis has been placed on experiments involving the Materials Experiment Assembly and Mid-deck experiments on the Space Shuttle.

The Microgravity Science and Applications Program Task Document covers the period of January 1988 - December 1988. The document includes research projects now being funded by the Office of Space Sciences and Applications, Microgravity Science and Applications Division, NASA Headquarters.

The Microgravity Science and Applications Division wishes to thank the Universities Space Research Association (USRA) and in particular Ms. Elizabeth Pentecost for her efforts in the compilation and publication of this report.

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II. TASKS

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A. GROUND BASED EXPERIMENTS

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1. ELECTRONIC MATERIALS

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Fundamentals of Electronic Crystal Growth

NASA Lewis Research Center
Thomas K Glasgow
In-House
October 1987 - Continuing

Growth and Characterization of Opto-electronic Crystals (W. Duval et al)

In cooperation with the Westinghouse Research and Development Laboratory the growth of opto-electronic halides is being examined. Attention is especially directed to determining growth rate and gradient effects as they are influenced by the occurrence of convection. These systems are especially appropriate for study because they are technically important and experimentally convenient. Some are not only transparent but different colored in the liquid qnd solid so the growth interface may be observed. Special transparent furnaces have been constructed to observe the growth.

Effects of Thermal Radiation (Duval and Kassemi)

The objective of this study is to mathmatically describe the influence of thermal radiative transfer in crystal growth processes, especially physical vapor transport. Initial work has been limited to radiation in the presence of transparent gasses and opaque crucibles. It has been shown that radiative transfer seriously distorts an otherwise planar growth interface. In future work the influence of non-transparent gasses will be studied.

Mathematical Modelling of Directional Solidification (Chait et al)

Using the approach of adapting to the greatest extent possible commercial codes rather than creating completely original software, a facility for in-house modelling of solidification processes has been created. Problems attacked have included the 3-M Diffusive Mixing of Organic Solvents, chemical vapor transport growth of silicon carbide, simulations of KC-135 and expendible launch vehicle experiments, and modelling of the GTE GaAs solidification furnace. Some of this work has become the subject of a supported proposal to model flight furnaces. Results of work already performed may be used to establish rapidly the time required in low-g to damp out pre-existing fluid flows. Numerous experimenters have requested modelling of their flight or preflight experiments.

Publications

Singh, N. B., Duval, W.M.B., and Rosenthal, B. N., "Characterization of Directionally Solidified Lead Chloride," J. Cryst. Growth, 89, 80-85 (1988).

Taghavi, K. and Duval, W.M.B., "Effects of Furnace Temperature Profile on the Interface Shape During Bridgman Crystal Growth," in *Proceedings of the 1988 National Heat Transfer Conference*, Volume 2 (H.R Jacobs, ed.), 1988, pp. 435-444.

Duval, W.M.B. and Jacqmin, D. A., "Dynamics of Two Fluids under Periodic Acceleration," in *Proceedings of the AIAA/ASME/SIAM/APS First National Fluid Dynamics Congress*, July 25-28, 1988.

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Presentations

Chait, A. and Arnold, W. A., "Transient Flow Behavior on Low-g Vehicles: a Numerical Case Study," *FIDAP Users Conference*, October 2-4,1988, Chicago, Ilinois.

Growth Kinetics of Physical Vapor Transport: Crystal Growth of an Opto-Electronic Material, Mercurous Chloride

Westinghouse R&D Center
Dr. N. B. Singh
NAS3-25274 (NASA Contact: W. Duval, LeRC)

Objectives: For the optical and acousto-optic devices refractive index of the material should be very uniform and the optical scattering should be low. This can be achieved by growing homogeneous, extremely pure and stress free crystals. For this reason, the crystal growth and transport behavior is being studied in transparent cylindrical ampoules under 1 g conditions. The present experiment should yield detailed insights into the relationship among convective phenomena, growth kinetics and, subsequently, the high quality of the crystal. The data from the ground-base experiment will be used to develop a flight experiment so that advantage of the microgravity environment to space can be used to enhance the optical homogeneity.

Research Task Description: The experiment is being carried out to define the effects of convective phenomena on the growth mechanisms and properties of the opto-electronic crystals grown by physical vapor transport. Mercurous chloride, which exhibits an anomalously slow sound velocity, a wide range of transparency, large birefringence, and very high acousto-optic diffraction efficiency is the material under study. Since the material is transparent and transports congruently we are investigating the relationship between growth parameters, convective behavior and morphology of the solid-vapor interface.

Progress to Date: Experiments to evaluate the effect of ampoule geometry on the Hg_2Cl_2 crystal growth rate are continuing. For a particular thermal environment the growth rate increases at higher values of a^2/h [where a is the radius of the ampoule and h is the transport length between the Hg_2Cl_2 source and crystal (sink)]. To examine the impurity layer build-up at the crystal-vapor interface, a detailed experiment was carried out in the following sequence. After first seeding the crystal and after growth started in the cylindrical part of the tube, the growth rate was carefully measured. (For this part of the experiment, the displacement length used to disturb the initial equilibrium was 0.353 cm). After a period of more than 120 hours, the pulling motor was switched on and crystal was grown at the speed of 0.5 cm/day. When more than two-thirds of the source material was consumed (transformed into single crystals), the motor was switched off to re-establish the equilibrium. After a few days, equilibrium was once more established. Then the crystal position was moved by 0.374 cm to destabilize equilibrium, and the growth rate was again measured.

During crystal growth Hg_2Cl_2 evaporates, sometimes leaving behind a residue. If there are volatile impurities co-evaporating with Hg_2Cl_2, they will form a so-called noncondensable impurity boundary layer near the crystal-vapor interface. In the first run described above, when the growth rate was measured the concentration of impurities was sexpected to be very low. For the second run the concentration of impurity was expected to be much higher, and the growth rate should have been much lower. The growth rates were identical for both segments of the experiment. Thus, there was no reduction in growth rate due to so-called stagnant impurity boundary layer at the interface of the growing crystal. This confirms that the purity of the starting material was very high. Similarly growth rate measurements are being taken for different thermal conditions to examine the effects of source temperature and hence the pressure.

Preliminary results show that data do not obey the H-K equation.

Publications

Singh, N. B., Glicksman, M. E., and Mazelsky, R., "Evaluation of Transport Conditions during PVT Growth: Mercurous Chloride System," Phys. Chem. Hydro., 1988 (in press).

Singh, N. B., Davies, D. K., Gottlieb, M., and Mazelsky, R., "On the Quality of Mercurous Chloride Crystal," J. Cryst. Growth, 1988 (submitted).

Presentations

Singh, N. B., "Growth of Mercurous Chloride Crystals in 1-g Conditions: Materials Processing in Space," *ASM Meeting*, Chicago, September 1988.

Solution Crystal Growth of Organic and Polymeric Materials for Nonlinear Optics Applications

NASA Marshall Space Flight Center
Dr. Marcus Vlasse
Dr. Donald O. Frazier
Dr. H. J. Caulfield, University of Alabama, Huntsville
Dr. S. C. Narang, Stanford Research Institute
In-House

Objectives: The major objective of the research is a thorough ground-based investigation of the basic aspects of solution growth of several representative diacetylenes and other organic compounds with nonlinear optical properties. Among the aspects to be studied are the influence of solvents, thermal gradients, concentration gradients, and, foremost, the influence of convective flows on the growth process and perfection of the crystal. Further problems to be studied are the evaluation of the influence of interface effects on defect incorporation into the crystals. Size and shape and growth habit of the crystal may play important roles in the growth process. Shape and size of growth cell may also be a significant variable to investigate.

The results of the study will be used to determine the advantages of growing such crystals in space under a reduced gravity environment. Furthermore, these findings will be used in solving current problems in the crystal growth of diacetylenes and other organic or biorganic substances with nonlinear optical properties.

Research Task Description: In this ground-based crystal growth experiment the plan of attack is the control of convection in a predictable manner as to be able to measure its effects and extrapolate to a microgravity environment.

The following basic steps will form the bulk of the research task.

A small scale growth experiment will be carried out using the evaporation method to produce crystals for further study and seeding. The bulk of the work will be performed using the controlled cooling method to produce the necessary supersaturation and induce nucleation and growth. Both seeded and unseeded runs will be introduced into this growth method to determine its effect on the growth faces. Reduction of convection will also be tried by the use of more viscous media such as gels, where the process is quasidiffusion controlled.

Detailed characterization of the crystals will be performed to determine the densities of chemical and physical impurities. Such characterizations will be performed mainly by spectroscopic means as well as microscopy and x-ray diffraction and topography. These data will be correlated to the various growth parameters and methods in order to determine the influence of such parameters on the quality and size of crystals. Some simple modeling of the growth process will be attempted to acquire a better understanding of the mechanisms involved.

Progress to Date: The apparatus and method have been set up for the preparation of a urethane substituted diacetylene (R-C = C-C = C-R), TCDU, not available in commerce. A certain amount of the pure compound, TCDU, has been synthesized. This method will be applied to other diacetylenes. The conditions (solvents, temperature, saturation, etc.) have been investigated for the growth of unseeded and seeded crystals of TCDU by

the evaporation method. Single crystals of TCDU, in the form of plates of good optical quality have been grown from ethyl acetate-acetone mixture at room temperature.

Work has been initiated to study the crystal growth of L-arginine phosphate (LAP), a very promising material for NLO applications. A certain necessary amount of LAP has synthesized and small crystals for seeding purposes have been grown. Further work has been carried out on the dependence of its solubility on temperature and pH in aqueous solutions. Saturation and speed of evaporation have been studied. Seeded growth experiments in various configurations have been performed. The results of this work so far have allowed us to grow single crystals of LAP of extremely good optical quality for further experimentation.

Publications

Vlasse, M., in Proceedings of the Polymer Investigations in Space Workshop, Huntsville, AL, March 17, 1988.

Heat Flow Control and Segregation in Directional Solidification

Massachusetts Institute of Technology
Professor August F. Witt
NSG-7645
(NASA Center Contact: R. Naumann, NASA HQ)
October 1, 1987 - September 30, 1988

Objectives: The objectives of this research are: (1) establishment of the limits of melt stabilization in Bridgman growth configuration by axial and transverse magnetic fields; (2) development of modular hot zone configuration that provides for quantifiable and controllable heat flow about the growth interface and for adjustable axial thermal gradients in the solid, liquid and vapor phase regions in Bridgman growth configuration; and (3) development of data base for the optimization of space growth experiments directed at the exploration of the potential of micro-g conditions for electronic materials processing.

Research Task Description: Growth and segregation experiments, involving Ga-doped Ge, CdTe, CdMnTe and CdZnTe, were carried out in a heat pipe based Bridgman system. To provide for stoichiometry control through the vapor phase and for melt temperatures in excess of 1050 degree C, an additional hot zone was added and the conventional high temperature heat pipe (maximum operating temperature of 1050 degree C) was replaced by an Inconel heat leveler system. Growth experiments with stabilizing axial magnetic fields of up to 30 kGauss were also conducted in THM configruation (Traveling Heater Method) for solution growth of HgCdTe. The growth facility was made available for research sponsored by DARPA and for a series of growth experiments on magnetic melt and solution stabilization conducted by staff from Texas Instrument Corporation.

The characteristics of macro-segregation in Ga-doped Ge grown with magnetic stabilization as well as an analysis of the density and distribution of stress induced line defects suggested a major modification of the heat pipe based hot zone. To minimize radial thermal gradients in the solid and to reduce point defect dynamics in the grown material, it is found necessary to provide for axial thermal gradients not presently attainable in the solid. Similarly, fluid dynamic considerations suggest application of controlled axial thermal gradients in the melt. A hot zone redesign is currently in progress.

Progress to Date: Using a two-inch gradient zone and quartz crucibles (15 mm ID) at lowering rates of 10 micron/sec, near planar growth interface morphologies were achieved during non-stabilized growth in the heat pipe based vertical Bridgman system; radial macro-segregation in Ga-doped Ge averaged at less than 3%; axial macro-segregation was in compliance with the Pfann equation, yielding a k(eff) of within 20% of k(o) (these experiments established the existence of pronounced laminar convective melt flows related to prevailing radial thermal gradients at considerable distance from the growth interface). Attempts to establish mass balance from the segregation data failed, either because of dopant loss during specimen preparation (etching) or because of partial GaO formation which renders the dopant electrically inactive.

Melt stabilization with axial and transverse magnetic fields of up to 3kG resulted in significant asymmetric radial dopant redistribution, but did not affect the macroscopic axial segregation behavior [k(eff[av]) unaffected]. These findings are consistent with a magnetic field induced restructuring of the convective melt-flow patterns and with the

appearance of related non-uniformities in the characteristics of the solute boundary layer.

Growth of Ga-doped Ge with melt stabilization by an axial field of 30kG yielded an axial dopant composition profile (for the first 20mm of growth) which is consistent with diffusion controlled dopant transport and in quantitative compliance with the Jackson-Tiller theory. Analysis of the remaining 70mm of grown crystal indicated complex macro-segregation behavior. The dopant concentration reaches a maximum at a growth distance of about 27mm from the original regrowth interface; it decreases with continuing growth and reaches its lowest level [appr. c(o)k(o)] at a distance of 90mm. The dopant concentration is found to subsequently increase exponentially over the last 10mm of growth. The data obtained from these experiments suggest the existence of a residual convection cell which allows for diffusion controlled build-up of the solute boundary layer. With continuing growth, i.e. changing aspect ratio of the melt, this convection cell contributes increasingly to dopant removal from the solute boundary layer and thus accounts at-large for the observed axial macro-segregation effects.

Publications

Witt, A. F., "Electronic Materials Processing and The Microgravity Environment", in *Proceedings of Symposium on Commercial Opportunities in Space: Role of Developing Countries* (K.E. Harwell and F. Shahrokho, eds.), AIAA Progress Series, 1987.

Matthiesen, D. H., Wargo, M. J., Motakef, S., Carlson, D. J., Nakos, J. S., and Witt, A. F., "Dopant Segregation During Vertical Bridgman-Stockbarger Growth with Melt Stabilization by Strong Axial Magnetic Fields," J. Cryst. Growth, 85, 557-560 (1987).

Carlson, D. J. and Witt, A. F., "Determination of Free Charge Carrier Distribution and Micro-Segregation of Dopants in n-Type GaAs," J. Cryst. Growth 91, 239-243 (1988).

Carlson, D. J. and Witt, A. F., "Quantitative Optical Determination of Segregation in Doped Silicon: A Comparative Analysis with Spreading Resistance Measurements," J. Cryst. Growth 91, 652-654 (1988).

Lin, C., Carlson, D. J., and Witt, A. F., "Growth Related Residual Strain in LEC GaAs," J. Cryst. Growth, 1988 (in press).

2. SOLIDIFICATION OF METALS, ALLOYS AND COMPOSITES

Immiscible Phase Incorporation during Directional Solidification of Hypermonotectics

University of Alabama in Birmingham
Dr. J. Barry Andrews
Dr. R. T. DeHoff, University of Florida

Objectives: One of the most potentially important factors in the development of immiscible alloys for engineering applications involves ways by which to increase the percentage of the immiscible phase incorporated into the structure above that of the monotectic composition. While this area is important, it remains almost completely unexplored in solidification research today. This research program is designed to provide an in-depth analysis of the manner in which the hypermonotectic phase is incorporated into the directionally solidified microstructure of immiscible alloys and should leas to the development of a model explaining reactions taking place at the growth front. It is hoped this research will result in a better understanding of ways in which to produce aligned composite structures in hypermonotectic alloys which contain a higher volume fraction of the immiscible phase than alloys of monotectic composition.

Research Task Description: Solidification behavior will be studied through the use of transparent analogue immiscible systems using a temperature gradient stage microscope. In addition to binary systems, transparent ternaries will be utilized in which the miscibility gap height can be varied in order to bring about a transition in morphology from an irregular to an aligned composite-like structure. Solidification will be carried out in a thin glass cell that will be horizontally oriented in order to minimize separation of immiscible phases due to gravity. The program will test the effect of growth rate, thermal gradient and miscibility gap height on the ability to incorporate the hypermonotectic liquid into the structure at the growth front.

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Studies of Containerless Processing of Selected Nb-Based Alloys

Vanderbilt University
Dr. Robert J. Bayuzick
Dr. M. B. Robinson, MSFC
NAG8-536
(NASA Contact: M.B. Robinson, MSFC)
July 17, 1985 - July 31, 1988

Objectives: Research is being conducted on the effect of containerless processing of alloys in a low-gravity environment. The primary goal is the better understanding of deep undercooling and its effect on microstructure and properties. The 100 meter drop tube at the Marshall Space Flight Center is being used to continue and extend work which has already been done to give a firm foundation of earth-based research.

Research Task Description: Previously, Nb-Ge alloys of compositions ranging from 13 to 35 atomic percent Ge were deeply undercooled. Undercoolings observed were as high as 25 percent of the liquidus temperature (Approximately 530 K). The microstructure and superconducting properties were extensively characterized. Work was also done on as focused on Pb-Pt and Nb-Si alloys ranging in composition from 10 to 32 atomic percent alloy additions. Undercoolings ranged from 15 to 27 percent of the liquidus temperature (absolute undercooling as high as 671 K). Investigations included scanning electron microscopy, x-ray powder diffraction, and measurement of the superconducting transition temperature. Higher composition Nb-Pt samples had undercooling limited by nucleation of the Nb_3Pt phase. Solute trapping is indicated at the lower compositions at the higher undercoolings. In Nb-Si, only the equilibrium phases have been noted.

Experiments on bulk samples of pure metals have also been conducted. Droplets were as large as 7 mm in diameter. Undercoolings up to approximately 23 percent of the melting temperature were obtained. These represent the largest absolute undercooling ever obtained in bulk samples. For example, the absolute undercooling in tantalum was about 740 K. The results were consistent and repeatable showing that these large levels of undercooling in large samples are readily obtained in containerless, microgravity experiments. It was clear that nucleation began near the free surface. However, such surface nucleation could be explained by surface energy arguments nor was it thought to proceed from surface oxides, particularly in the case of molybdenum, niobium, and tantalum. Thermal gradients in the drop samples are thought to be responsible for the surface nucleation.

Progress to Date: Since January 1988, work on Nb-Pt alloys and Nb-Si alloys has continued and experiments on Nb-Ni alloys were begun. A model was developed to determine dendrite solidification velocities from the shape of the recorded recalescence peaks for undercooled samples. Using this model, solidification velocities were measured for the undercooled Nb-Pt samples. For the Nb-Pt 16-18 atomic percent samples, solidification velocities were determined to be 4 m/s which is neat the solute trapping limit of 8 m/s as calculated by an Aziz-type analysis. In these samples, partitionless solidification occurred due to the rapid solidification and resulted in samples which were single phase Nb_3Pt. The undercooled samples thus solidified by nculeation and growth of Nb_3Pt directly from the melt, in contrast to both the equilibrium phase diagram and the as-cast samples.

Drops of Nb-Si alloys ranging from 15 to 30 atomic percent Si were electromagnetically levitated in a back-filled He environment. Once molten they were

undercooled to different degrees, released from the coil, and subsequently splat-quenched with the hammer and anvil technique. Analytical Transmission Electron Microscopy, Scanning Electron Microscopy, and X-ray Diffraction techniques were applied to characterize the alloys. The analyses revealed metastable microstructures. In the eutectic range, a two-phase fine-grain structure of Nb and Nb_3Si phases with grain sizes as small as 40 nm were found. Also lamellar and rod-like eutectic microstructure with spacings of 40 nm were observed. A metastable eutectic composed of Nb and Nb_5Si_3 was identified in a number of cases. Near the 25 atomic percent Si concentration the Nb_3Si phase was formed congruently and, with sufficient undercooling prior to splatting, no evidence of the peritectic reaction was seen. A different solidification pathway occurred when the Nb_5Si_3 phase precipitated first in which case the Nb_3Si peritectic phase was absent from the microstructures. In this case, the end result was primary Nb_5Si_3 with the metastable Nb plus $NbSi_3$ eutectic already described. Amorphous phase was observed from 17 to 21 atomic percent Si over relatively large volumes.

Nb-Ni alloys from 45 to 75 atomic percent Ni were similarly processed. Large amounts of amorphous phase wqas produced in almost the whole range of compositions. Significant extended solubility of Nb in Nb_3Nb and Ni in NiNb was observed. Regular and irregular eutectic microstructures were found of extremely fine morphology.

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Solidification Processing of Dispersed Phase Reinforced Mg Alloy Composites under 1-G and Microgravity Conditions

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Professor Julian Szekely
NAG3-308
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Objectives: The objective of this research is to conduct experiments and to perform calculations regarding the production of metal matrix composites by stirring solid particles into melts and by the subsequent solidification of the melt-solid suspensions thus produced. The main motivation for this work is twofold: (1) metal matrix composites are thought to play a key role in the construction of space-based structures; and (2) the solidification of melt-solid suspensions could be ideally studied in a microgravity environment, because under these conditions sedimentation would be greatly reduced.

Research Task Description: The theoretical work is concerned with establishing the fundamental factors that govern the introducton of the solid particles into melts, establishing the fundamental factors that govern the suspension of the solid particles in melts, the definition of the rheology of melt-solid suspensions, and the definition of factors required for production of uniform distribution of dispersoids.

Progress to Date: It is found that the wetting characteristics of the melt-solid system play a paramount role in determining whether a solid can be successfully introduced into the melt; the extent of agitation plays only a minor role. It is found that intensive agitation is crucial for suspending solids in melts, whenever there is a density difference between them. The melt-solid suspensions are found to be non-Newtonian with a strong power 1/wa type behavior. A mathematical model has been developed to represent the flow behavior of these systems.

Experiments on mixing of particulates led to the development of a new technology which eliminated the entrapment of atmospheric gases normally associated with vortex mixing. Patents have been filed. An experimental approach has been initiated for improvement of the distribution of particles through high shear rate mixing and controlled solidification rates. Patents have been filed. An experimental apparatus has been constructed for measuring the viscosity of semi-solid sluries isothermally and in temperature gradients. Basic rheology properties have been measured for Al and Mg matrix sluries with SiC particulates.

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Braze Metal Flow in Planar Capillaries

Massachusetts Institute of Technology
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NSG-7645 (NASA Co ract: R.K. Crouch, NASA HQ)
October 1, 1986 - September 30, 1988

Objectives: There is considerable evidence suggesting that braze joints could be as strong as weldments if the defects at the joint could be eliminated. The goal of the proposed research is to measure the interface morphology of a braze alloy advancing in a planar capillary, to quantify the forces that drive the flow and that produce the instabilities, and to attempt to stabilize the interface by the addition of an appropriate gradient.

Research Task Description: Two methods were proposed at the beginning of this study by which this flow may be measured: (1) solder flow in glass capillaries and (2) braze alloy flow in copper capillaries using infrared thermography. A braze alloy flows faster into a thin capillary, presumably due to the relatively greater ratio of interfacial tension to inertial forces. By imposing a geometric gradient in the capillary spacing, it may be possible to stabilize the interface. Another known variable in the speed of braze metal flow is surface roughness, where greater roughness behaves the same as a relatively larger interfacial tension. This suggests that transverse grooves, or gradients in surface roughness may stabilize the braze front. Yet another important variable is the composition of the solid being wetted. Gradients in the thickness of active metal coatings might provide a stabilizing factor in this system. Since buoyancy forces are known to be important in brazing, the orientation of gravity may have an effect on the stability of the interface. Studies of metal flow in different orientations may provide insight into the physics of braze metal flow. Finally, brazing in microgravity may well stabilize the interface merely by reducing the Rayleigh number of the system.

Progress to Date: These tests with glass slides, and then the construction of the vacuum IR thermography chamber have consumed the initial period of work. Current research is assessing whether we can measure solder or braze alloy flow in planar capillaries. This second experimental method is now being used to study interface stability under thermal, geometric and compositional and roughness gradients. In addition, recent tests have shown that a low mwlting metal alloy can be used to wet the glass capillaries, permitting direct observation of metal flow at low temperatures.

Model Immiscible Systems

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Barbara R. Facemire
William K. Witherow
In-House

Objectives: The objective of this research is to identify effects that wull contribute to phase separations and to final ingot micro- and macro- morphology in solidification of monotectic alloys. The approach is to use organic systems that can serve as metal models thereby taking advantage of transparency for optical applications and ambient phase transitions for ease of study.

Since the study of transparent immiscible systems has been an important method of investigating metallic monotectic alloys, it is important to suitably generalize from observations in the model systems to metallic monotectics. Recent work on model transparent systems has focused on homogeneous solution component and surface interactions to assess subsequent effects on macrosegregation during fast quenches through the monotectic temperature. To the extent that the existence of the miscibility gap itself can serve as a "signature" for certain thermodynamic characteristics of the solution, for example, deviations from ideality, it is appropriate to determine as completely as possible the key thermodynamic parameters for at least one such model system. From the model, study directions may arise for specific metal systems, which could result in better control of ingot microstructure and macrostructure.

Research Task Description: Methods have involved the use of differential scanning calorimetry (DSC), desnsitometry, holographic techniques and other optical methods (Fornier transform, infrared spectroscopy) to exploit phase transition ambience and solution transparency to determine fundamental aspects of solution dynamics during solidification processes. It is important at some point in time to assess on a molecular level, the processes that occur during equilibration and, ultimately, solidification, in some specific system. Therefore, intermolecular interactions gain significance through the study of solution ideality and spectroscopy. All methods exploiting this approach describes the tasks of this project.

For the succinonitrile-water system, differentil scanning calorimetry (DSC) and densitometry show a significant effect of homogeneous solution equilibration temperature on surface induced composition shifts. These shifts influence monotectic reaction onset in fast-quenched small-volume samples. Relative effects between high and low equilibration temperatures appear to vary with respect to the isopycnic temperature. Equilibration of succinonitrile-rich solutions above the isopycnic temperature in hydrophilic containers generally result in "less undercooling" and larger heat release than equilibrations below the isopycnic temperature. No such ordering exists in similar solutions fast-quenched in hydrophobic containers. We postulate from partial molar volume calculations, that at the isopycnic temperature, homogeneous succinonitrile-water systems tend to behave ideally. Above and below this temperature, solute-solvent aggregates differ significantly and if strong solute-container affinities are present, these aggregates will influence radial composition profiles with significant specificity.

Fourier Transform Infrared (FTIR) spectroscopy of a succinonitrile-benzene solution has been successful in giving 1-cm^{-1} resolution spectra at 1- to 1.5 m

penetration depth into the bulk phase through a zinc selenide attenuated total reflectance crystal. Determination of preferential wetting properties on zinc selenide with respect to succinonitrile-rich and benzene-rich phases, by use of a contact angle goniometer shows that at room temperature, benzene-rich phases preferentially wet zinc selenide. There are differences in homogeneous succinonitrile-benzene surface spectra apparent upon heating and equilibrating at temperatures ranging from near monotectic to critical. These differences are currently under analyses to help define equilibration temperature effects on surface aggregation in these miscibility-gap type systems.

Further on-going investigations use Raman and resonance Raman spectroscopic methods to determine preferred bulk-phase cluster profiles in succinonitrile-based systems at different temperatures. Dr. John Hall at the Dolphus E. Millian Science Research Institute is performing ab initio self-consistent field calculations to determine the optimized geometries of trans and gauche conformers of succinonitrile and the degree of hydration for each conformer. From the models established by this approach, the group at MSFC will perform normal coordinate analyses on complexes using reasonable force fields to duplicate vibrational frequencies observed in the surface FTIR amd bulk-phase Raman spectroscopic analyses.

Progress to Date: Differential scanning calorimetry of near monotectic succinonitrile-water solutions, fast quenched in hydrophilic and hydrophobic DSC pans, indicate by degree of undercooling, that there may be significant dependence of final ingot microstructure on pre-quench equilibration temperature. Partial model volume determinations from density data, along with DWC data, suggest the nature of temperature dependent component associations from 20 C to 55 C in homogeneous solutions. The undercooling profile in a hydrophilic container may be explained in terms of solution composition shifts arising from the Gibbs Surface excess. The evidence shows that temperature dependent preferred component aggregates may modulate surface composition gradients. Similar effects may be present through intermetallic compound formation in metallic monotectic alloys.

Additionally, through holographic and shadowgraph techniques supplemented by two wavelength holographic measurements, two primary sources of fluid motion are identified in the transparent analogs. One is the existence of thermal and compositional gradients in the single phase bulk melt, and the second is related to either phase separation and the formation of droplets in the two phase regions (immiscibility gap and the solid-liquid interface), or, to the existence of the monotectic temperature. In both bases buoyancy forces play a role. When deposits are formed, thermocapillary effects may dominate and fluid flow will influence mass transport. This compositional profile in the melt differ significantly from purely diffusive profiles.

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Gravitational Effects on Liquid Phase Sintering

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October 1, 1986 - September 30, 1988

Objectives: The results obtained from this research will be aimed at improving the understanding of microstructural development, mechanical properties, component shape retention, and dimensional stability benefits that may be realized from low gravity sintering of materials that form a liquid phase during processing. These studies will provide information on the gravitational contribution to slumping, distortion, and microstructural coarsening that occur during liquid phase sintering. Gravity-induced settling due to density differences between the solid and liquid phases produces components with a high degree of distortion, as well as an inhomogeneous microstructure. Gravity's effect on settling has been seen to depend on both configurational energy and kinetic considerations. The increase in the number of particle contacts at the bottom of a compact has been seen to influence the coarsening rate, and subsequently the mechanical properties. By isolating the role that gravity plays, critical conditions for successful liquid phase sintering may be established.

Research Task Description: Tungsten heavy alloys (W-Ni-Fe) have been chosen for this study due to the large density difference between the liquid and solid phases. Several test geometries varying in size and shape are being used to determine how and when slumping occurs, and how this affects the microstructural development of the material. The test matrix contains cmaterials varying in the amount of solid present at the sintering temperature. A number of different sintering times and temperatures have been chosen to study the development of the microstructure. The degree of slumping and distortion is measured for each sample. Quantitative measurements ofthe changes in volume fraction of solid, grain size, contiguity, and connectivity between the top and bottom of sintered compacts are made. Finally, the mechanical properties are measured to determine how they are affected due to gravity-induced changes. Coupled with experimental results are theoretical calculations of the amount of settling to be expected based on configurational energy changes that are occurring throughout the compacts. This must be combined with kinetics to determine the overall effect gravity has on the microstructural development of liquid phase sintered materials.

Progress to Date: The last nine months have centered on quantifying the experimental results for the range of alloys sintered at one temperature for various times. Major shifts in the microstructural features are seen to occur at long sintering times, when the contiguity and connectivity undergo reductions. A possible change in the dominant coarsening mechanism may be occurring, and this change may be gravity aided. A statistical analysis of the results provided a first model for grain growth that includes a contiguity and volume fraction of solid correction. A computer model was created that incorporates gravitational settling combined with energy increases due to grain shape accommodation. Th grain shape effect is necessary to facilitate the higher amounts of solid. Currently, a model is under development which combines thermodynamic and kinetic considerations. Separate experiments consisting of samples made up of very low percentages of solid have been incorporated into this study to help in developing the

model. Experiments have also begun using the matrix conditions but sintering at a higher temperature to determine the effects of liquid viscosity and accelerated microstructural changes on gravitational settling and subsequent component properties.

Publications

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Solidifcation Fundamentals

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1983 - Continuing

The objective of this research program is to obtain a fundamental understanding of gravitational effects during solidification of metals and alloys. Experimental work has been in several categories: experiments in support of a Space Shuttle experiment on macrosegregation, crucible wetting experiments, directional solidification experiments delineating regimes of dendritic and cellular growth, casting of Ge-Si alloys,and bulk undercooling to elucidate micro- and macrosegregation behavior. This task has also partially supported the construction and operation of a dual heat pipe leveled directional solidification furnace with quenching capability.

Publications

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Studies of Crystallization of Freely Suspended Spheres and Shells

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Dr. W. Q. Rhim
NAS8-34605
(NASA Contact: V. Fogle, MSFC)
January 1987 - January 1988

Objectives: To utilize earlier results on crystallization of bulk supercooled melt and supersaturated solutions in high and low g to elucidate processes in crystallization of spheres and shells. Applications lie in the following areas.

(1) Atmospheric Sciences

(a) Freezing of supercooled cloud and rain drops and production of secondary ice particles; growth of frozen drops from the vapor as single polycrystals.

(b) Crystallization of haze and sea salt solution droplets under conditions of low humidity to give sing;e/polycrystals.

These topics are relevant to precipitation processes and artiificial stimulation of precipitation, with relevance to drought problems and the radiation balance of the earth's atmosphere with applications to problems of long and short term climatic charge.

(2) Metallurgy and Materials Science

Crystalline spheres and shells may be utilized in the manufacture of sintered materials and light weight (low mass) but high strength materials for space station construction. The crystallinity and impurity distribution in the component spheres or shells is of major importance in determining the overall mechanical and thermal properties of these highly specialized materials. Similar considerations apply to shells for laser fusion studies.

(3) Crystallization of proteins is of importance in producing single crystals for structure determination. Isolated solution droplet for crystallization may provide an important technique for producing large crystals under low-g controlled environment conditions.

(4) Interaction of growing crystals with solid surfaces gives rise to changes of growth habit, growt velocity, and significantly influences the microstructure of the completely solidified melt. This work has application in general understanding and characterization of castings, freeze dried materials in biology, and also may give fundamental information on how crystals grow under stressed conditions.

Research Task Description: To produce spherical droplets and shells of known composition; to achieve uniform supercooling/supersaturation of these systems under controlled conditions and nucleate these systems in a controlled manner. The subsequent crystal growth to be examined in real time, and to investigate the influence of supercooling and solute concentration of the crystal structure and overall mechanical properties of the sphere or shell. The ideas developed would be usedto design experiments forfurther idealization of crystallization udner conditions of low gravity and to design experiments for utilizing opportunities of the low-g of the shuttle/space station.

Progress to Date: A system has been designed for suspension of 1 cm drop in a liquid sandwich of two mutually immiscible liquids such that the drop can be supercooled (or supersaturated) uniformly in absence of contact with solid interfaces. Nucleation is achieved by insertion of a small single crystal of known orientation into the drop, and the subsequent crystal growth examined by VCR. Growth rates and habits have been examined for water suspended between paraffin and CCl_4. The results show that crystals grow singly to the periphery and then stop, failing to grow around the interface. This is in complete contrast to growth in a solid crucible or on a solid surface where curved stressed crystals grow in contact with the solid, and multinucleation may occur. Water expands on freezing to produce symmetrical spikes on each side of the drop, growing at 90 to the "a" axis growth rate.

Growth experiments have been carried out on growth on walls of different thermal properties which show faster growth rates on high thermal conductivity substrates. Supercooled water films (as soap bubbles) have been crystallized to show that growth is uniform around the sphere, and independent of direction; it appears that the stresses in the soap film itself bend the growing crystal to retain maximum growth direction independently of the curvature.

Spherical solution drops have been suspended in the elextrostatic suspension at JPL developed by Dr. W.Q. Rhim. These droplets evaporate and nucleate as single crystals, which grow as individuals out of contact with any surface. This shows that under specific conditions in one-g, single crystal nucleation can be achieved from solution as it can from pure melts. Previous studies of convective motion in one-g and lack of convective motion in low-g show that specific criteria can be established for the formation of single and polycrystalline drops (and probably thick shells) for crystallization in both low and high g.

Publications

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The Development and Prevention of Channel Segregation During Alloy Solidification

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NAG3-560 (NASA Contact: Dr. R. L. Dreshfield, LeRC)
July 15, 1986 - July 14, 1989

Objectives: The objective of the research is to identify the conditions under which channel segregation occurs during the solidification of alloys over a freezing range, and to make comparisons between materials having different physical properties, summarized by the Lewis and Prandtl numbers.

Research Task Description: During solidification of materials which have a significant freezing range, segregation channels can develop in the mushy zone of the partially solidified dendrite array. These channels extend approximately vertically as pencils of solute rich material and in alloy castings they can be regarded as serious defects. The cause of such channel formation is a density inversion between interdendritic and bulk liquid which is brought about when the solute(s) is less dense than the solvent component, e.g., tin in lead, carbon in iron, water in ammonium chloride. The incidence of channels depends upon the growth conditions, temperature gradient and growth rate, and upon the alloy composition which influences th permeability of the mushy zone. The onset of convection which leads to channel formation originates at or just ahead of the dendritic growth front and depends upon the thermal, solutal diffusivity ratio (Lewis number) and the ratio of the kinematic viscosity to thermal diffusivity (Prandtl number).

Progress to Date: A fully automated gradient furnace has been assembled with facility for digital recording of information from multiple thermocouples: this is being used to vary the interdendritic spacing which is thought to influence the onset of channel convection. In addition to th data which has been assembled for metallic (lead base) systems and the aqueous ammonium chloride system, observations of channel formation are being made in organic materials (succinonitrile-ethanol) and ionic salts (sodium fluoride - sodium chloride), these having different Lewis and Prandtl numbers.

Publications

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Whisker Growth Studies Under Conditions Which Resemble Those Available on an Orbiting Space Laboratory

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NAG 3-642
(NASA Contact: L. Westfall, LeRC)
(Expires March 4, 1989)

Objectives: The objectives of this research task are: (1) determination of advantages, disadvantages, and special circumstances attendant upon annulment of earth's surface gravity during nucleation and growth of metal crystals (especially whiskers), (2) investigation of significance of electric currents which are a concomitant of such growth in applied electric fields used to levitate the whiskers, and (3) develop short-duration growth experiments suitable for flight in NASA aircraft microgravity laboratories.

Research Task Description: Originally all whisker growth was performed using the hydrogen reduction of metal halides within an apparatus which provided both an applied electric field (for annulment, or reversal, of earth gravity) and the ability to record the accompanying electric currents. This apparatus also provided routine visual observation of the growth process. Later, many other configurations, including growth under very high substrate pressure, growth at very low gas pressures (down to the onset of glow discharge in the apparatus), growth from small spheres of halides, and growth from vacuum deposited thin films of metal halides.

The work outlined in the original proposal has been largely accomplished and has resulted in what might be called a breakthrough in the understanding of the basic nucleation process for these type whiskers. It has also provided clear indications that a short duration growth experiment (of the order of 6 seconds) is possible: such an experiment could easily be flown on one of the NASA aircraft and forms part of a new proposal for continuation of this grant.

Sufficient data have been accumulated to strongly indicate that (a) the electric currents are <u>not</u> directly related to the growth; moreover, purely electrical augmentation of the growth does not occur, (b) the beneficial effects of the levitating electric field are essentially purely mechanical, and (c) that convection in the molten growth substrate may play a very significant role: namely, to suppress growth by destroying a thin surface film which recent data shows to be the active site for nucleation of the whiskers. The findings to date have effectively changed the research task to that of following up on the new perspectives provided by the breakthrough, performing a preliminary in-flight experiment, and formulating an adequate theory to describe the genesis of whiskers within the metal-supersaturated molten substrate. It now appears that unlimited uniaxial growth of fine single crystals are a distinct possibility: worth an additional year or two of effort.

Progress to Date: Progress may be summarized as follows: (1) Levitating fields <u>do</u> effect the growth;(2) Factor of 10 improvement in growth length of Cobalt whiskers; (3) Experimental basis for new theory for genesis of reduction grown whiskers; (4) Development of "hydrogenless" growth; (5) New technique using annular growth patterns to explore for ideal growth conditions; (6) Demonstration of extremely high density growth (in excess of 10^6 whiskers per cm^2); (7) Development of very fast growth experiments (suitable for NASA aircraft labs); (8) Establishment of a connection between substrate convection and whisker growth.

Publications

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Presentations

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Structure of Nickel and Iron Aluminides Prepared by Rapid Solidification and Undercooling

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NAG8-475
(NASA Contact: E.C. Ethridge, MSFC)
August 1, 1984 - July 18, 1988

Objectives: The objective of this investigation is to obtain a basic understanding of the complex solidification structures found in the nickel-base aluminides during rapid solidification and undercooling.

Research Task Description: The Ni_3-Al, Fe-Al-C and Fe-Ni-Al-C systems have been selected for study. Particular interest lies in fcc-like metastable structures in the Fe-Al-C and Fe-Ni-Al-C systems which can be revealed by rapid solidification. Rapid solidification studies are carried out in an arc hammer apparatus and by melt spinning at controlled and variable cooling rates. Undercooling experiments are conducted in the 100 m drop tube at Marshall Space Flight Center. Structural studies use x-ray diffraction and transmission electron microscopy techniques.

Progress to Date: The quench-rate metastable structure-dependence as well as the effect of composition was examined in the Fe-Ni-Al-C system x-ray diffraction and electron diffraction studies showed the quench-rate dependence of metastable structures present in the rapidly solidified samples. The effect of composition (i.e. presence of silicon) was also verified to play a major role in the kinetics of phase formation.

Rapidly solidified samples of Fe-Al-C alloys were also prepared. A combination of x-ray difraction, electron diffraction, and image analysis was employed to verify th presence of metastable structures (fcc and ordered fcc). A phase field was estsblished to show the various phases present for different compositions. So far, no quench rate dependence was found on samples 35-70 microns thick.

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Crystal Growth by Two Modified Floating-Zone Processes

University of Wisconsin
Professor Sindo Kou
NAG8-705 (NASA Contact: S.L. Lehoczky, MSFC)
March 1, 1988 - February 28, 1991

Objectives: Floating-zone crystal growth under microgravity, though essentially free from natural convection, can still suffer from undesirable Marangoni convection. The objectives of this study are to: (1) reduce Marangoni convection and the thermal fluctuations it produces in microgravity floating-zone crystal growth, and (2) to enhamce diameter control and surface quality of crystals grown under microgravity by floating-zone.

Research Task Description: To effectively reduce this convection while at the same time help produce single crystals of uniform diameter and smooth surface, two modified floating-zone processes are being studied. The first of the two processes uses a ring heater in contact with the melt surface and the second a sheet heater immersed in the melt, both (heaters) with careful temperature control during crystal growth. The first part of the proposed work is the direct observation of Marangoni convection in the two processes, using a transparent material of high Marangoni number. The second part is the characterization of the two processes, with emphasis on effects of process variables and search for optimum growth conditions. The third part is the computer modelling of the two processes and the experimental verification of the computer models.

Progress to Date: Direct observation of Marangoni convection is being carried out using a molten zone of $NaNO_3$, which is transparent and in which Marangoni (rather than natural) convection dominates. An optical system consiting of a He-Ne laser and cylindrical lenses is being set up to produce a light cut through the molten zone, to allow visualization and analysis of convection through a video camera and a high-resolution image processing system. Work on computer modeling of Marangoni convection in the molten zone is being continued, using body-fitted orthogonal curvilinear coordinates.

Metallic Glass Research in Space

Jet Propulsion Laboratory
Dr. Mark C. Lee
NAS7-918
October 1, 1985 - continuing task

Objectives: The objective of this research is to develop a space experiment to acquire thermodynamic properties of bulk metallic glasses over the entire undercooling region, with emphasis on the temperature region inaccessible by terrestrial techniques.

Research Task Description: Ground-based precursory experiments will be designed and performed in such a manner that all the critical parameters for the space experiment will be defined and validated. A logical approach to achieve this goal should include the following ground-based tasks: (1) development of a novel contactless calorimetry technique for specific heat measurements over the entire undercooling region of a bulk metallic glass sample; (2) measurements of specific heats and crystallization kinetics to precisely define the optimal candidate systems for the space experiment; (3) conceptualization of the data analysis technique for the space experiment; and (4) feasibility study of the metallic glass space experiment module.

Progress to Date: A noncontact true temperature measurement technique using a laser pyrometer has been further developed to actively support the Drop Physics Module (DPM) flight hardware development program. In addition, two flight proposals are being prepared in response to the Announcement of Opportunity (AO) currently solicited by NASA.

Publications

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Lee, M. C., "The First Chinese Student Space Shuttle Getaway Special Program," *1988 GAS Experimenter's Symposium*, NASA/KSC, September 26-30, 1988.

Levitation Studies of High Temperature Materials

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Professor John L. Margrave
Shankar Krishnan
George P. Hansen
Robert H. Hauge
NAG8-612

Objectives: This research is a proposed three-year program which is designed to expand capabilities for doing levitation research by moving into the microgravity of space. It will allow the establishment of highly reliable thermodynamic and other properties of elements like silicon and boron in both solid and liquid states, without the risk of container contamination. Also, the phenomenon of super-cooling, nucleation and kinetics of crystal growth which are so important in semiconductor development can be studied without the interference of gravity, vibrations, container impurities and dust.

Research Task Description: Studies will be conducted which yield monochromatic spectral and hemispherical emissivities of liquid boron and liquid silicon at various wavelengths and temperatures. Also, the densities of the liquids will be determined by a photographic technique at various temperatures.

Progress to Date: Research efforts over the past year have been focussed on: (1) design of optimum coils of levitation of good conductors (Hf, HfC, etc.) and of poor conductors (B, Si, SiC, etc); (2) design and construction of an optical system for photographic determination of liquid metals; (3) design and construction of a system for high-speed photography of levitated objects - solid or liquid; (4) development of the background mathematical equations from which surface tensions and viscosities can be related to experimental oscillations of liquid droplets; and (5) development of techniques for determining emissivities of liquid metals over wide ranges of temperature and wave length.

During the second year of the project, tasks to be accomplished are: (1) complete studies of emissivities (Pd, Pt and Ir studies are in progress); (2) measure high-temperature thermodynamic properties of Hf, HfC and ZrC (solids and liquids-in progress); (3) measure high-temperature thermodynamic properties of B, C, Si and SiC (in progress); and (4) determine surface tensions and viscosities of liquid transition metals--Cu, Ni, Ti, Fe, etc.-in progress.

Publications

Hansen, G. P., Krishnan, B., Hauge, R. H., and Margrave, J. L., "A New Method to Determine Temperature and Emissivity of Liquid Metals at Elevated Temperatures," Trans. Met. Soc., 1988 (accepted).

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Containerless Processing of Undercooled Melts

University of Wisconsin, Madison
Professor John H. Perepezko
NAG3-436
(NASA Contact: H. de Groh, LeRC)

Objectives: The main objective of the research is to evaluate the undercooling and resultant solidification morphologies in the containerless technique of drop tube processing. The degree of liquid undercooling attainable in a laboratory scale (3m) drop tube can be altered through the variation of processing parameters such as melt superheat, droplet size, and environmental conditions. At a certain level of undercooling in a given molten sample, nucleation and growth kinetics between equilibrium and metastable phases compete in the microstructural development. This solidification behavior is evaluated through metallography, thermal analysis, and x-ray examination with a heat flow model of the processing conditions.

Research Task Description: Process parameter effects on undercooling and structural evolution are critical in the evaluation of drop tube processing. Under controlled conditions variations of particle size and processing environment has resulted in a multitude of microstructural developments in Ni-53 at% Nb. For example, under constant enviromental processing conditions, a reduction in droplet size increased the fraction of droplets which undercooled to the glass transition temperature. This behavior is attributed to the isolation of internal nucleants and the increase in cooling rate which accompanies a reduction in droplet size. In addition, at a constant size distribution, an increase in the thermal conductivity of the gas environment leads to an increased fraction of amorphous powder due to increased cooling rates. Moreover, a change in gas can alter the catalytic potency of the surface through chemical reaction to allow nucleation and subsequent growth of a different metastable phase. A more thorough identification of these effects is currently being continued to understand the competition during phase selection in undercooled liquids during containerless processing.

With a qualitative understanding of the solidification behavior in drop tube processing, a quantitative analysis of the thermal history is required. Direct thermal measurement in drop tube powder processing is difficult, but thermal histories can be evaluated through alternate techniques. Millimeter size droplets of various Fe-Ni alloys can be dropped from a specified superheat, and the falling distance before solidification can be measured by adjusting a copper quench plate height. Undercooling levels at these various heights can then be calculated through a Newtonian heat flow analysis. The solidified microstructures together with the determined undercooling can then be compared to a calculated metastable phase diagram to check the validity of the result. A compilation of size ranges and alloy compositions has led to the development of a processing-microstructure map to delineate regimes of structural/morphological evolution in drop tube processed Fe-Ni. Work to expand upon this analysis is being pursued currently.

Publications

Perepezko, J. H. and Allen, W. P., "Processing of Undercooled Melts," in *Proceedings of Third International Colloquium on Drops and Bubbles,*" Monterey, CA, September 1988 (submitted).

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Ro'e of Gravit, on Macrosegregation in Alloys

University of Arizona
Professor D. R. Poirier
C.F. Chen
J.C. Heinrich
NAG3-723
(NASA Contact: A. Chait, LeRC)
April 1986 - April 1989

Objectives: The major objective is to develop comprehensive convection/solidification computer codes to model macrosegregation in alloys that freeze in a dendritic mode. The finished codes could be used to design experiments to study the effect of a low gravity environment on macrosegregation in binary alloys. It is also anticipated that the codes would be used to assist engineers in designing or controlling commercial casting processes in which convection is driven by gravity.

Research Task Description: In order to model macrosegregation phenomena in alloys which freeze dendritically, a quantitative analysis of solute redistribution is absolutely necessary. Hence appropriate forms of the mass, momentum and energy equations must be selected to predict each of these transport processes in solidifying castings. In addition to predicting macrosegregation variations across a casting or from its bottom to top, major emphasis is on modeling the intricate convective phenomena responsible for localized defects, often called "freckles", which are particularly troublesome to practitioners. Emphasis is on multi-diffusive convection, which is thought to be the cause for the "freckles". When combined with thermodynamic data for gas-forming reactions, the basic solidification model can be extended to predict the conditions when interdendritic porosity forms or, indeed, to predict the avoidance of such a defect. Because the overall program deals with defect-avoidance, it is expected that practitioners will derive significant benefit from the research.

Progress to Date: A part of the early effort in the program was in collecting and evaluating physical and thermal properties. Such data must be quantitatively analyzed so that extrapolations to the solidification temperature range can be made with confidence.

Linear stability analyses and nonlinear calculations for a fully convecting system have been done for a liquid layer above a stationary porous medium of constant porosity. This was done as a first attempt to simulate convection in directionally solidified castings comprising an all liquid zone above the mushy zone. Results show that the one-equation models, such as that used by Bennon and Incropera, Beckermann and Viskanta, and Voller and Prakash, may underestimate the resistance of the interface. More recently the model has been extended to treat alloy solidification more realistically by considering an all liquid zone above a mushy zone moving with a prescribed solidification velocity and with a variable volume fraction of liquid (i.e., "porosity") in the mushy zone that is consistent with solidification thermodynamics. As of this writing, this more realistic model is being used to simulate solidification at 1g and at reduced values of g. Such computations can be used by other NASA investigators who are designing space processes and/or experiments.

Another accomplishment was the development of a code to treat steady-state directional solidification of a binary alloy in the form of a circular cylinder. The code is particularly useful to examine the sensitivity of the interdendritic convection and macrosegregation to a slight curvature of the nominally horizontal isotherms in the

mushy zone of a DS casting. This geometry was inspired by experiments at NASA-LeRC designed to study macrosegregation in Pb-Sn alloys as preliminary work for space experiments.

Publications

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Microgravity Solidification Processing of Monotectic Alloy Matrix Composites

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Dr. Y. Shiohara
NAG8-084 (NASA Contact: P.A. Curreri, MSFC)

Objectives: The objectives of this research are to: (1) utilize the combination of the microgravity environment and the unique wetting characteristics of monotectic allots to produce microstructures not obtainable elsewhere; (2) provide understanding of the basic principles governing the solidification of monotectic alloys; (3) conduct a theoretical and experimental study of monotectic allot solidification in the constrained spaces between ceramic fibers; and (4) understand the role of gravity vector in the solidification of monotectic alloys.

Research Task Description: The proposed three year research program was to proceed along both experimental and theoretical fronts. The program is designed as a coordinated, cooperative effort between MIT, NASA, ABEX, and United Technologies Research Center.

Experiments are to be conducted on several monotectic alloys, beginning with Al-In. Solidification is to be both with and without Al_2O_3 or SiC ceramic particles or fibers. Initial experiments are to be conducted in normal gravity in a Bridgman-type furnace built specifically for the project. The furnace is designed so that the key solidification variables, thermal gradient and crystal growth rate may be varied independently.

After obtaining baseline data at MIT, solidification experiments will be conducted under sub-orbital microgravity conditions. A mathematical modelling study is to be conducted in parallel with the experiments. Diffusion and fluid flow equations will be solved to predict the solidification morphology as a function of processing parameters. Theoretical results will be of great value in planning and interpreting experimental work.

Progress to Date: A unidirectional solidification apparatus has been constructed. This apparatus is capable of attaining the high temperature gradients and low growth rates necessary for regular monotectic growth. The growth rate and the temperature can be independently controlled. The entire apparatus can be rotated to study the effects of the direction of the growth vector relative to the gravity vector on the solidification behavior. Gradients to 400K/cm have been achieved.

Samples of the monotectic composition have been directionally solidified at various temperature gradients and growth rates. This was done to check results of our experiments against those found in the literature as well as provide conditions for our modelling work that were not reported in the literature (i.e. extremely slow growth rates. The samples were found to obey the reported $\lambda^2 R$ = constant relationship).

Composite samples with a matrix composition of Aluminum-50 wt% Indium, reinforced with SiC and Al_2O_3 particulates have been formed. These samples were made by pressure infiltration of the molten alloy into a powder compact which has been packed to a uniform density. Using the same method, we are currently making samples which have different matrix compositions. We are also currently experimenting with

trying to fund a processing method for the Al-In-non-metallic fiber composites. We expect to be able to form these composites in a similar manner to that in which the particulates composites are formed, however, we are running into difficulties in obtaining a method in which the non-metallic fibers are well aligned and/or regularly spaced.

Following the Jackson-Hunt model for eutectic growth, we have been able to predict the constant in the $\lambda^2 R$ = Constant relation from material parameters. A value of C 9.8 x $10^{-17} m^3$/sec is predicted from the model, which is in very good agreement with the empirically reported value of C = 4.5 x $10^{-16} m^3$/sec. The radius of the L_2 fiber tubes in the regular monotectic structure was also compared to the critical particle radius for entrapment in an Uhlmann, Chalmers and Jackson particle pushing analysis. The critical radius for entrapment is seen to be slightly lower than the observed radius. We have also studied the theoretical composite (regular monotectic structure - as opposed to the metal matrix composites above) stability range with respect to growth rate and thermal gradient.

Publications

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Containerless High Temperature Property Measurements

Midwest Research Institute
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NAG8-465
(NASA Contact: L. Gardner, MSFC)
January 1988 - January 1989

Objectives: The objective of the research is to do advanced containerless processing and materials research at high temperatures in space. In this way, the production and processing of very pure and high quality forms of important ceramic, superconducting, semiconducting, very hard, very strong, and other useful kinds of materials may be achieved.

Research Task Description: New techniques adaptable to in-space work have been developed in earth-based research and the limits of earth-based containerless experiments are advanced and defined so that good choices for in-space R&D can be made. Methods of experimentation include gas jet and electromagnetic (EM) levitation, laser heating with or without EM heating, and laser induced fluorescence or mass spectrometric measurements of vapor and ambient gas concentrations. Non-contact temperature measurement is achieved by optical pyrometry. A new, absolute method for liquid specimen emittance measurement is under development.

Progress to Date: Research to date has employed a combination of techniques for containerless experiments, including gas jet, and electromagnetic levitation, EM and CW CO_2 laser heating, laser induced fluorescence (LIF measurements of ambient and vapor atom concentrations and temperature measurements at high temperatures on materials of high purity and materials that react with containers.

Some of the results provide promising directions for continued earth based research. For example, optical properties of very pure single crystal sapphire were obtained at temperatures up to the melting point of Al_2O_3 (2327K) and accurate vapor pressures were measured for LaB_6, a material for which no non-reactive container material exists at the experimental temperatures (up to 2500K). Although there are other materials for which similar experiments would provide important results, such experiments would not further develop capabilities for space-based R&D because they can be completed entirely on earth. Stable gas jet levitation of laser heated liquids has not been achieved and it appears that acoustic positioning is the preferred method for containerless in-space R&D on liquids that are poor electrical conductors. For electrical conductors, EM levitation and heating is possible on earth and in space. In a low-gravity environment, the combination of radiant heating with EM positioning techniques promises very wide application. Low power, very high frequency levitators may be developed to extend in-space EM positioning of relatively poor conductors of electricity.

Electromagnetic levitation was used to achieve containerless conditions. CW CO_2 laser heating was used to heat the specimen above the minimum temperature achieved by EM heating. This improved levitation stability by allowing independent control of the levitation force and temperature. Vapor analysis is by laser induced fluorescence and, for experiments carried out in a vacuum, by mass spectrometry. An optical pyrometer is used to determine apparent specimen temperature, which can be corrected to the true temperature if the specimen's spectral emittance is known.

Study of the Influence of Gravity on the Solidification of Ceramic Metal-Matrix Composite Material

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Dr. D. M Stefanescu

Objectives: The objective of this research is to study the factors affecting the distribution of oxide particles (Al_2O_3, SiO_2) and carbide particles (SiC) in nickel-aluminum alloys and aluminum nickel alloys during directional solidification at 1-g and low-g, in order to obtain information conducive to a process for obtaining uniform, homogeneous distribution of ceramic particles throughout a Ni-Al metal matrix.

To achieve this objective, it will first be necessary to generate information and reach some basic understanding ofth mechanism of particles behavior in front of a moving solid-liquid interface (particle pushing and particle entrapment).

Using directional solidification to control the solidification rate, and microgravity to alter the influence of surface tension, and collision rate, coupled with ground based experiments, the science and processing technology to produce metal-matrix composite materials (particulate composites) will be further developed.

Research Task Description: The nickel-aluminum system was selected to provide the metal matrix because of the fact that this system exhibits two eutectics, a high temperature one at 85.7%Ni (1387 C) and a low temperature one at 6.1%Ni (640 C). The high temperature eutectic alloy is used in temperature applications. The low temperature eutectic alloy is to be used as a model, to allow for better understanding of the phenomena.

The composites were prepared by melting and degassing Al-6.1%wt.pct. Ni alloys and adding SiC particles in a mechanically stirred vortex. The stirrer used was made of graphite. In most cases, 5-10 vol% of particles of either 1 to 7 m or 50 to 150 m size were added. Due to segregation of the SiC particles in the melt, the volume fraction obtained in cast samples differed.

The samples were directionally solidified in alumina crucibles both in the University of Alabama's Bridgman-type furnace and in a similar furnace aboard NASA's KC-135 aircraft, flying on parabolic trajectories, under gravity levels varying from 10^{-2} to 1.8 g. The parameters varied were furnace translation rate, temperature gradient and gravity level. After solidification, the samples were cut longitudinally and their microstructures examined. The particle distribution in the samples were obtained as a measure of interparticle distance.

Progress to Date: Several ground based and KC-135 experiments were carried out. The ground based experiments mostly documented the pushing/entrapment behavior of the particles by the melt interface. It was demonstrated that particles can be entrapped or pushed by the moving solid/liquid interface, if adequate solidification rates and temperature gradients are used. The experiments on KC-135 demonstrate that high gravity, high volume fractions, of particles or high effective viscosity of the liquid favors the engulfment of the particles by the melt interface. Solidification in low gravity seems to defloculate the SiC particle agglomerates while opposite results are obtained when solidifying under high gravity. Intercellular spacings are found to be higher in low-gravity solidification as compared to high gravity solidification.

Publications

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Theoretical Studies of the Surface Properties of Liquid Metals and Semiconductors

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Dr. Zhiqiang Wang

Objectives: The principal objective of this research is to calculate the surface tension and other thermodynamic properties of a number of liquid compound semiconductors and liquid metals as functions of temperature and impurity composition, thereby gaining a better understanding of the forces underlying convection in a low-gravity environment.

Research Task Description: This project will initially involve calculating the surface tension of liquid CdTe as a function of temperature and concentration, using empirical two- and three-body potentials fitted to the bulk cohesive energy, lattice constant, and alloy heat of formation. The surface tension is calculated using a direct Monte Carlo evaluation of the work required to create surface. Calculations are carried out on a CRAY X-MP-4 of the Ohio Supercomputer Center. Other thermodynamic properties such as bulk and surface specific heat and surface atomic arrangements, will also be calculated. Calculations contemplated for the near future include (1) extension to other compound semiconductors, such as GaAs and (2) development of molecular dynamics programs suitable for treating kinetic properties such as viscosity and ionic mobility. The influence of gravity and electric fields on these properties will be estimated.

Cellular/Dendritic Solidification of Binary Alloys in a Positive Thermal Gradient

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NCC-395
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September 1987 - September 1989

Objectives: The objective of this research is to study the development of cellular/dendritic microstructures during directional solidification of binary metallic model alloys in a positive thermal gradient. Important differences are predicted among cell/dendrite growth models under conditions where the gradient of constitutional supercooling is small. Conflicting experimental behavior of primary arm spacing and dendrite tip radius has been reported in the literature in this growth regime.

Research Task Description: Pb-Sn and Pb-Au alloys will be partially directionally solidified and quenched to retain the cell/dendrite tip morphology and solutal distribution in the melt. Important microstructural features: the cell/dendrite tip radius, tip temperature, liquid composition at dendrite tips and the primary arm spacing will be measured as a function of processing variables, such as, growth speed, thermal gradient and alloy composition. The experimentally observed behavior will be examined against theoretical predictions from cellular/dendritic growth models.

Progress to Date: A vacuum controlled atmosphere casting apparatus has been designed and fabricated to prepare metallic alloy speciments with in-situ incorporated thermocouples. The directional solidification furnace in the Microgravity Materials Science Laboratory at NASA/Lewis is being used to prepare the directionally solidified and quenched Pb-10 wt% Sn, Pb-5 wt% Sn and Pb-8 wt% Au specimens. Steady state thermal profiles have been established during growth. However, initial evaluation shows that considerable macrosegregation is present along the length of the directionally solidified specimens. It suggests that thermosolutal convection may make it impossible to obtain a steady state solutal profile during growth of these ground-based processed specimens. Thermal and solutal steady state growth is assumed in all the dendrite growth models.

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Containerless Studies of Nucleation and Undercooling

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Dr. Eugene H. Trinh
In-Center

Objectives: The long term research objectives are to perform experimental and theoretical studies to determine the achievable limits of undercooling using acoustic and other means of sample levitation and positioning, to study the characteristics of heterogeneous nucleation of levitated samples, and to measure the physical properties of significantly undercooled melts. Specially designed ultrasonic levitators operating in ground-based laboratories as well as in the NASA KC-135 aircraft are used to levitate, melt, undercool, and solidify 0.1 to 3 mm specimens of pure metals and alloys (Ga, In, Sn, Al), as well as organic compounds (O-Terphenyl,Succinonitrile), and low melting glasses. Non-perturbing measurement techniques for the surface tension, viscosity, density, and sound velocity are being used and refined to probe the physical state of undercooled levitated melts. Methods for the determination of other thermophysical properties such as the specific heat and thermal diffusivity are being investigated or developed.

Research Task Description: A final set of data has been obtained for the surface tension of undercooled pure Indium in an Argon atmosphere. The highest values of surface tension which have been obtained agree with published data extrapolated from the stable liquid range. A detailed experimental study of the slow fluid flows induced within the melts levitated in ground-based laboratories has shown that residual fluid motion exists in most of the samples undergoing levitation undercooling in 1 G.

Progress to Date: Statistical data are being gathered in an attempt to correlate the observed limit of undercooling to the acoustic pressure amplitude required for levitation and to the internal flow magnitude. Finally, studies of the solidification process of undercooled (and maybe hypercooled, in some instances) succinonitrile have resulted in the observation of dendritic growth. New laboratory experimental capability is being assembled to provide for a higher hydrostatic pressure (20 Bars), higher temperature (1000 C), and high vacuum outgassing facilities.

Publications

Trinh, E. H., Marston, P. L., and Robey, J. L."Acoustic Measurement of the Surface Tension of Levitated Drops", J. Colloid Interface Sci. 124, 95 (1988).

Ohsaka, K. and Trinh, E. H.,"On Dendritic Growth in Undercooled Melts", J. Cryst. Growth, 1988 (in press).

Ohsaka, K. and Trinh, E. H.,"Partitionless Solidification in Undercooled Droplets", Scripta Metall., 1988 (submitted).

Trinh, E. H. and Robey, J. L. "Acoustic Cooling/heating Device for Microgravity", NASA Tech Briefs July/August 1988 (patent pending).

Presentations

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Influence of Convection on Microstructure

Clarkson University
Dr. William R. Wilcox
NAG8-480 (NASA contact: Peter Curreri, MSFC)
June 1984 - December 1987
Renewal expected for August 1988 to 1991
(NASA contact: Frank Szofran, MSFC)

Objectives: The long term goal of this research is to gain an understanding of the influence of microgravity on the microstructure of the MnBi-Bi eutectic.

Research Task Description: David Larson and Ron Pirich of Grumman showed that directional solidification of the MnBi-Bi eutectic in space or on Earth with a magnetic field applied results in a fiber spacing 1/2 of that obtained by solidification on Earth without a magnetic field. We had shown previously that the microstructure is unaffected by temperature gradient and that the microstructure responds more quickly to a change in freezing rate than the freezing rate changes in response to a change in ampoule translation rate.

Progress to Date: Computer computations were carried out for the influence of convection on the compositional field in front of lamellar and fibrous eutectics with a planar interface, and the resulting effect on microstructure. Experimental results with spin-up/spin-down (Accelerated Crucible Rotation Technique) gave good agreement with predictions for lamellar eutectics. However buoyancy - driven convection was calculated to be too weak to noticeably influence the microstructure.

Electrochemical experiments showed that spin-up/spin-down causes large fluctuations in mass transfer, and therefore in heat transfer and freezing rate during solidification. Decantation experiments showed that the MnBi fibers project for large distances into the melt during solidification, and that they infrequently branch. Elevated temperature fracture of samples also revealed little branching of the MnBi fibers. Even without spin-up/spin-down, large temperature oscillations occurred in the melt. The amplitude of the temperature oscillations decreased as the solid-liquid interface was approached; the frequency increased as the melt volume decreased.

Spin-up/spin-down experiments were also performed on the lead-tin eutectic. While the lamellar spacing was unaffected under the conditions used, spiralling of the microstructure depended strongly on rate of rotation of the ampoule. Rotation also caused one end of the ingot to be lead rich while the other was tin rich.

Following is the work statement for the renewal of this project, to run from August 1988 to August 1991.

1. Perform theoretical work for the cooperative solidification of MnBi-Bi two phase mixtures, as follows:

    a. Influence of the Soret effect on the average composition vs. distance of off-eutectic mixtures in the absence of convection.

    b. Influence of convection on the microstructure of off-eutectic mixtures using a linear velocity gradient in the melt.

c. Influence of volumetric change of solidification on microstructure.

d. Influence of convection on microstructure when the MnBi fibers project out in front of the bismuth matrix.

2. Tabulate the physical properties of all eutectics solidified in space to see if these point the way to an explanation of the different effects of microgravity on microstructure; volume fraction eutectic, eutectic temperature, densities of the pure constituents, density changes upon solidification, etc.

3. Measure the Soret coefficient and the diffusion coefficient for Mn-Bi melts of eutectic composition.

Modelling Directional Solidification

Clarkson University
Dr. William R. Wilcox
NAG8-541
(NASA Contact: Frank Szofran, MSFC)
September 1985 to January 1989

Objectives: The long term goal of this project is to develop an improved understanding of phenomena of importance to directional solidification, to enable us to explain and predict differences in behavior between solidification on earth and solidification in space.

Research Task Description: Experiments on organic compounds showed that in contrast to recent computer models, the convection in a vertical Bridgman-Stockbarger ampoule is usually not axisymmetric and may vary with time. If the temperature in the furnace increases with height the convection may be greatly surpressed. On the other hand, if the temperature decreases with height the convection may be vigorous. (Prior theoretical models had built assumptions of steady state, axisymmetric flow, and a constant heater temperature into their initial equations.) Experiments are underway to determine the influence of convection on compositional homogeneity of directionally solidified organic compounds. A computer code was developed to calculate the radial variation of composition dur to a curved interface in the absence of convection.

Progress to Date: KC-135 experiments showed that liquid in a non-wetted cylindrical ampoule does not pull away from the ampoule wall in low g, as has been proposed to explain the observation that ingots solidified in space often have diameters smaller than their containing ampoules. Rather the liquid either separated into separate columns or formed one or more bubbles along the walls. Theory predicted that a bubble is unstable beyond a critical size. This prediction agreed with experiment. Likewise experiment and theory agree on the fraction of the flat walls in a triangular ampoule that are in contact with the melt (the melt pulls away from the corners only). The new transparent solidification apparatus ("polymer solidification furnace") built for MSFC could help resolve the mystery of the reduced diameter ingots obtained in space.

Theoretical analyses were done on the diffusional decay of compositional variations in a crystal during the period it is cooling to room temperature. Decay of striations is favored by a slow freezing rate, a small period for the composition variations, a small temperature gradient and a large diffusion coefficient in the solid. Under some realistic conditions striations decay within a few wavelengths, while under other conditions they may persist for a long distance. Thus the compositional inhomogeneities observed in a grown crystal are not necessarily indicative of those produced at the growth front.

Apparatus and procedures were developed to determine the influence of spin-up/spin-down and of freezing rate fluctuations on the perfection of directionally solidified InSb-GaSb alloys. Mullard laboratories in Southampton, England have shown both increased homogeneity and much larger grain size caused by spin-up/spin-down during solidification of HgTe-CdTe alloys. The mechanism responsible for increased grain size is unknown; the possibilities proposed include a more convex interface shape, scrubbing of nuclei from the walls, and meltback during each cycle of the spin-up/spin-down. In one apparatus spin-up/spin-down is being used, while in another identical apparatus large electric currents are used to cause period meltback of the interface

without convection. We are determining the interface shape, microstructure and compositional variations as a function of the growth conditions.

Publications

Gray, R. T., Larrousse, M. F., and Wilcox, W. R., "Diffusional Decay of Striations," J. Cryst. Growth, 1988 (in press).

3. FLUID DYNAMICS AND TRANSPORT PHENOMENA

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Thermo-Diffuso Capillary Phenomena

Lewis Research Center
Dr. R. Balasubramaniam
Dr. A.T. Chai
Dr. L.H. Dill
Mr. J.B. McQuillen
In-House

Objectives: The objective of this program is to advance the understanding of capillary phenomena including convection processes driven by surface tension effects which result from temperature and/or concentration gradients.

Research Task Description: The research involves three areas of interest: (1) thermocapillary motions of bubbles and droplets in a thermal gradient in a host fluid. In this area analytical studies have been performed including effects of inertia and convection. Numerical simulations for a bubble have just been completed for a range of Reynolds and Marangoni numbers. Transient motion of bubbles and drops and the effect of a slowly adsorbing and desorbing surfactant on bubble migration are being theoretically analyzed. An experimental investigation of thermocapillary effects in droplets is being pursued in an immiscible density-matched system; (2) contact angle measurements, where experimental studies are being conducted to determine the feasibility of utilizing the constant curvature of liquid-vapor interface encountered at low Bond number (i.e. low gravity) conditions to provide accurate measurements of contact angles. Special sample container and rig have been designed and fabricated and testing will begin soon; and (3) two-phase flow under microgravity, where a precursor study is planned to examine the effects of changes in conduit geometry (diameter contractions, expansions and bends) on two phase flow behavior such as flow patterns, mixing, pressure losses and loading on the supports.

Progress to Date: Research in this area included studies on thermocapillary convection and oscillation in a thin liquid layer and on the natural convection due to thermal gradients in rectangular enclosures. These studies have neem concluded and the results have been documented in the publications cited below.

Publications

Balasubramaniam, R. and Chai, A. T., "Thermocapillary Migration of Droplets: An Exact Solution for Small Marangoni Numbers," J. Colloid & Interface Sci. 119, 531-538 (1987).

Hasan, M. M., and Balasubramaniam, R., "Thermocapillary Migration of a Large Vapor Slug in a Tube," J. Thermophys. & Heat Transf., 1988 (accepted).

Kassemi, S. A., "High Rayleigh Number Convection in Rectangular Enclosures with Differentially Heated Vertical Walls and Aspect Ratios Between Zero and Unity," NASA TM- 100277, 1988.

Balasubramaniam, R. and Wilkinson, R. A., "Electrohydrodynamic Migration of Charged Droplet in an Insulating Fluid," in *Proceedings of First National Fluid Dynamic Congress*, Cincinnati, July 1988 (in press).

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Presentations

Dill, L. H. and Balasubramaniam, R., "Unsteady Thermocapillary Migration of Bubbles," Third International Colloquium on Drops and Bubbles, Monterey, CA, September 1988.

Hydrodynamic Instability as the Cause of Morphological Breakdown during Electrodeposition

Massachusetts Institute of Technology
Professor R.A. Brown
Professor D.R. Sadoway

Objectives: The objective of this work is tc understand the origins of surface roughening during the course of electroplating. Specifically, the central question is the role of hydrodynamic instability in the electrolyte in initiating and promoting morphological breakdown during the process.

Research Task Description: The work combines experiments to measure flow characteristics and resulting surface structures with calculations of critical physical parameters. The study is unique in several respects. The causes of morphological breakdown during electrodeposition has never been the subject of a systematic study that seeks to investigate the problem under conditions where the kinetic processes are clearly defined. Specifically, this study is being conducted in a physical model system that will permit the observation of simple electrocrystallization under conditions of strict mass transfer control. Furthermore, this study is unique in its attempt to combine experimental observations with mathematical models of complex fluid flow behavior originally developed for the analogous solidification problem in crystal growth. There are, however, important differences between solidification and electrocrystallization, and these differences serve as the basis for this comparative study.

The experimental program measures the time for the onset of buoyancy driven convection by laser interferometry. As well as the determination of the time constant, this technique reveals the characteristic spatial dimension of the electrolyte circulation cells. These data are compared with scanning electron micrographs of the surface of the electrodeposit in order to test the hypothesis that in the absence of hydrodynamic instability morphological breakdown is completely avoided. Furthermore, the development of techniques for direct in situ measurement of surface quality is being pursued.

Because the experiments focus on mass transfer controlled electrocrystallization microgravity has an important role to play here. In field of solidification it has been shown that during crystal growth gravity greatly influences the critical concentration for the onset of convective and morphological instability. It is expected that the experiments in this electrocrystallization study will proceed so as to prepare for in flight testing of the theory and calculations.

Progress to Date: The first major accomplishment of this research has been the discovery of a physical model system in which simple electrocrystallization occurs under conditions of strict mass transfer control. It was extremely difficult to identify such a system because the database is almost bare in two important respects: (1) the physical properties of appropriate aqueous electrolytes and and (2) mass transfer controlled electroplating.

The physical model system is the electrodeposition of silver onto silver in acid nitrate solutions. This chemistry was chosen because silver is not complexed in these solutions, i.e., silver exists in these solutions as discrete Ag^+ ions, and the deposition of silver onto silver is known to have a relatively high exchange current density. Even so, strict control of electrolyte composition is imperative in order to confine the system to

the desired kinetic domain. Specifically, one must actively regulate pH to avoid nitrate decomposition onto nitrite. As part of the present study this nitrate nitrite decomposition reaction was studied by voltammetry and Raman spectroscopy. Results indicate that if the pH of the electrolyte solution moves outside a very narrow range a film forms on the electrode surface inhibiting the metal deposition process. The consequence of this is that the rate controlling step shifts from simple mass transfer control to a form of activation control where an activation step associated with puncturing the film limits the overall deposition rate. Under activation control it is impossible to test the central thesis of this study. Thus, control of the microchemistry at the electrode/electrolyte interface is critical and the research to date has identified the appropriate compositional interval.

Electrodeposits have been produced under a variety of parametric settings. It has been demonstrated that already in the very early stages of deposition, i.e., during the first few seconds, the morphology possesses the features characteristic of the kinetic control mechanism.

Experimental and Theoretical Studies of Wetting and Multilayer Adsorption

National Bureau of Standards
Dr. J. W. Cahn
Dr. R. F. Kayser
Dr. M. R. Moldover
Dr. J. W. Schmidt
April 1977 - continuing task

Surface Tensions in Refrigerants

Surface tensions in two environmentally acceptable refrigerants (R134a and R123) were made using capillary tubes of several bore sizes in a high pressure cell. The resulting capillary rise data when combined with densities yielded surface tensions of high accuracy over a temperature span in excess of 100 K. Auxilliary boiling ponts and densities were also determined. These replacement refrigerants will be used as the national phases out presently used refrigerants that are harmful to the environment.

The Structure of a Fluid Interface

The structure of the liquid-liquid interface in four mixtures and one ternary mixture were measured using ellipsometry. Measurements from all five mixtures scaled consistently to the same dimensionless constant (the elliptical thickness divided by the refractive index difference and the correlation length. Physical models and methods of calculating ellipticities from these models were developed to interpret the results. The best model currently is a capillary-gravity wave model combined with a density profile model in which there are no adjustable parameters.

Systematics of Wetting

Five first-order wetting transitions have been located at the vapor-liquid interface for a series of alcohol + fluorocarbon mixtures. Contact angles of fluorocarbon-rich pendant drops (suspended at the vapor-liquid interface) were measured for the series. In addition surface tension for the fluorocarbon-vapor, alcohol-vapor, and liquid-liquid interfaces were measured using a modified Du Noüy ring technique.

Interfacial Tension in the Critical Region

The importance of reliable estimates of interfacial tension in systems considwered for materials processing in space is increasing. Methods for accurately predicting interfacial tensions in CO_2+hydrocarbon mixtures near have been developed in collaboration with Dr. J. Rainwater (NIST Boulder). These methods ar presently being extended to new environmentally acceptable refrigerants.

Wetting Layers on Solid Substrates in a Gravitational Field

When two partially miscible phases of a binary liquid mixture coexisting in contact with a solid substrate and in a gravitational field a thin film can develop next to the solid substrate. In addition to dispersion forces which stabilize the film it can happen that electrostatic forces will stabilize thick films under certain conditions, namely when the film consists primarily of polar molecules and the other phase consists primarily of non-polar molecules. A new derivation is presented which calculates the

thickness of such films stabilized against gravity.

Wetting in Fluorocarbon + Alcohol Mixtures

Surface tensions and liquid+liquid interfacial tensions were measured in a series of alcohol + fluorocarbon mixtures using direct surface tension measurements by a du Nuoy ring technique combined with three-phase contact angle measurements on pendant flurocarbon droplets. Five first-order wetting transitions were located and follow an approximately linear trend $t \propto n$ where t is the reduced temperature and n is the alcohol chain length.

Publications

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Moldover, M. R. and Rainwater, J. C., "Interfacial Tension and Vapor-Liquid Equilibria in the Critical Region of Mixtures," J. Chem. Phys. 88, 7772 (1988).

Sengers, J. V. and van Leeuwen, J.M.J., "Capillary Waves of a Vapor-Liquid Interface near the Critical Temperature," Phys. Rev. A, 1988 (submitted).

Convection and Morphological Stability during Directional Solidification

National Institute of Standards and Technology
Dr. S. R. Coriell
Dr. G. B. McFadden
Dr. J. R. Manning

Objectives: The general aim of this task is the study of the fluid flow, solute segregation, and interface morphology which occur during directional solidification, including effects of gravity and microgravity. Control of solute segregation during solidification will allow preparation of materials with optimum properties. Space flight experiments, designed to determine cellular wavelengths as a function of growth conditions, are planned in collaboration with J. J. Favier and D. Camel of the Centre d'Etudes Nucleaires de Grenoble utilizing the directional solidification furnace being developed by the MEPHISTO project.

Research Task Description: The main focus of this task is the interaction of fluid flow in the melt with the crystal-melt interface, and the solute segregation which is controlled by interface morphology and fluid flow. In the absence of fluid flow, the conditions for morphological instability during directional solidification of an alloy at constant velocity are well established. However, for processing conditions for which instability occurs, the resulting non-planar interface morphologies (cellular or dendritic) are an active area of current research. Although progress has been made in calculating cellular morphologies, the question of wavelength selection is unresolved, and definitive experiments (without convection) are needed to provide guidance and a benchmark for theoretical developments. Numerical algorithms to calculate cellular morphologies in the absence of convection are being developed. Linear stability analyses of the effect of various types of fluid flow on the onset of morphological instability are carried out in order to delineate the role of convection. Fluid flow in the melt is calculated by a time-dependent, two-dimensional finite difference algorithm as a function of the gravitational acceleration. This allows prediction of the differences between experimental results obtained on earth and in space. The effect of time-dependent gravitational accelerations (g-jitter) is studied both by linear stability analyses and by numerical solution of the nonlinear fluid flow equations. Since the MEPHISTO furnace uses electrical pulsing and Seebeck measurements, research on the effect of electrical currents on the solidification process and solute redistribution is also underway.

Progress to Date: For processing conditions near the onset of morphological instability, a three-dimensional weakly nonlinear analysis has been carried out to second order in the interface deformation, taking into account the effects of differing thermal conductivities of crystal and melt, and anisotropy of the crystal-melt surface tension. The analysis suggests the pattern of the cellular interface morphology near the onset of instability. Linear stability analyses are being applied to a number of problems associated with the morphology of the crystal-melt interface. The effect of a two-dimensional stagnation flow on double diffusive and morphological instability has been calculated. The flow stabilizes the system with respect to perturbations that are perpendicular to the flow; the double diffusive instability is significantly reduced by the flow. We plan to investigate the role of buoyancy on morphological stability during directional solidification vertically upwards for the case of rejection of a heavier solute at the crystal-melt interface. Preliminary results indicate that for certain

alloys a long wavelength instability can occur.

The effect of a constant electrical current on the morphological stability of planar interface during directional solidification of a binary alloy at constant velocity has been investigated. Electromigration of solute, differing electrical and thermal conductivities of crystal and melt, Joule heating, and thermoelectric effects (Peltier, Seebeck, and Thomson effects) modify the conditions for morphological stability.

During solidification of an alloy at constant velocity, solutal convection can occur. The effect of this convection on the solute segregation in crystals grown by vertical directional solidification of binary metallic alloys or semiconductors has been calculated using finite differences in a two-dimensional, time-dependent model that assumes a planar crystal-melt interface and rigid sidewalls. For constant gravitational accelerations, as the solutal Rayleigh number is varied, multiple steady-states and time-dependent states may occur. The bifurcation from the quiescent state may be subcritical or transcritical, depending on the aspect ratio of the container. Calculations have also been performed for a gravitational acceleration that is uniform in magnitude with its direction rotating uniformly.

Publications

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McFadden, G. B., Coriell, S. R., and Sekerka, R. F., "Effect of Surface Tension Anisotropy on Cellular Morphologies," J. Cryst. Growth 91, 180-198 (1988).

Modeling of Coalescence, Agglomeration, and Phase Segregation in Microgravity Processing of Bimetallic Composite Materials

University of Colorado, Boulder
Professor Robert H. Davis

Objectives: The primary objective of the research is to develop a comprehensive model of the various gravity-related, surface tension-related and other contributing mechanisms that lead to phase segregation of liquid phase miscibility gap and powder/melt bimetallic composite materials. Droplet and particle collision rates will be computed by considering hydrodynamic and interparticle attractive interactions; these collision rates will be used in a population dynamics model in order to predict the size distribution evolution of the dispersed phase. An important novel feature of the research is the incorporation of spatial variations or inhomogeneities into the population dynamics model in order to predict local accumulation of the dispersed phase, such as at the walls of the container for preferential wetting materials, at a hot spot in the interior or at the end of the sample for thermocapillary-dominated processes, or at the bottom of the container for gravitational sedimentation-dominated processes.

Research Task Description: The primary goal of the research is to predict and optimize the final microstructure or grain-size distribution of the second phase as a function of the processing conditions (gravity level, cooling rate, initial level of mixing, etc.) and material properties (surface tension, composition, densities, viscosities, etc.). The primary significance of the research is that it will yield a better understanding of the dominant mechanisms behind the observed phase segregation of space-processed and earth-processed bimetallic composites, and will lead to improved methods for bimetallic materials processing.

Theory of Solidification

NASA Lewis Research Center
Prof. Stephen H. Davis
NAG 3-747
October 14, 1986 - October 13, 1989

Objectives: The research concerns the effort to understand on a quantitative level how various factors affect the morphology of a solidification front of binary materials. These factors include buoyancy-driven convection with and without Soret diffusion, forced flow, phase-change convection, crystal and kinetic anisotropies and effects of bounding surfaces. The central theme is the understanding of the phenomena through the study of the instability behavior of the appropriate coupled systems.

Research Task Description: The research entails the study of coupled systems using analytical methods, with the aim of identifying new mechanisms of behavior and new effects.

Progress to Date: In the past year we have identified a new mechanism of instability in directional solidification, viz. flow-induced morphological instability, driven by non-parallel flows over solidifying interfaces. We find that the rotation of a crystal during solidification produces flow that could be responsible for the creation of rotational striations.

We have shown when solutal-buoyancy effects are important, heat losses from the sidewalls of ampoules can be responsible for curved crystal/melt interfaces whose shapes are largely independent of the radial segregation. Thus "engineering" the interface to be nearly planar may not guarantee that the radial segregation is small. We have analyzed directional solidification near the absolute stability limit and have been able to predict the presence of hexagonal nodes as stable, three-dimensional patterns, at the onset of morphological instability. We have also shown how these nodes break down into two-dimensional cells as the degree of constitutional undercooling is increased. We have generalized Ivantsov dendrite solutions to systems with N-components, that can display cross-diffusional coupling (Soret and Dufour effects) and that are subject to arbitrary potential flows. These dendrites can have general shapes: paraboloids, hyperboloids, ellipsoids, etc.

Publications

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Disorder-Order Transitions in Colloidal Suspensions: Computer Simulations and Experimental Observations

Princeton University
Dr. P. G. Debenedetti
Dr. W. B. Russel

Objectives: The goal of this work is to understand the rate and mechanisms of formations of ordered, crystalline structures in concentrated colloidal suspensions, experimentally and computationally. Experiments will be aimed at developing a technique for the production of macroscopic single crystals by the application of periodic electric fields. From a scientific viewpoint, the dynamics of this phase transition has been largely unexplored. Practically, understanding the disorder-order transition is of importance in areas such as ultrafiltration and ceramics fabrication from submicron particles via colloidal processing routes.

Research Task Description: Our most important experimental task is to grow macroscopic single crystals by applying a periodic electric field at the bottom of a container to induce order in the first few layers and provide a pattern for subsequent crystal growth. This will be done by evaporating conducting electrodes on a quartz plate, interdigitating two combs of electrodes, and applying opposite voltages to each. This will produce a periodically modulated electric field gradient whose profile extends a short distance into the sample volume. The resulting force on the colloidal particles will induce order which is periodic in two directions, and either commensurate or incommensurate with the preferred three-dimensional structure of the resulting crystal. This technique, analogous to epitaxial film growth, may enable the creation of perfect three-dimensional single crystals of large size.

Computationally, we plan to modify our existing Brownian Dynamics code to include space-and-time-dependent systematic external forces, such as the periodically modulated electric fields to be used in the epitaxial growth experiments. This will allow us to study the kinetics and mechanism of field-induced growth of crystalline phases in real time and at the particle level. All simulations will be performed on supercomputers, using our own vectorized code.

Progress to Date: We have performed settling experiments with silica spheres bearing grafted octadecyl chains and dispersed in cyclohexane. We have found that settling of the particles under gravity produces iridescent sediments only if the theoretically predicted rate of crystallization exceeds the rate of sediment growth. For sufficiently small particles or low initial volume fractions (both of which reduce the rate of sediment growth), a polycrystalline sediment is formed, with closed-packed, isotropic crystallites near the bottom giving way to columnar crystals above.

Computationally, we have studied the growth of ordered regions within initially disordered, metastable, high volume fraction colloidal suspensions. This was done by simulating the dynamics of ca. 500 particles within a periodically bounded computational cell. The evolution of order was detected by Voronoi tessellation, and mechanisms for crystalline growth were identified. The growth of face-centered nuclei was found to occur via the formation of a predominant, topologically related precursor type of nucleus. We have studied the disorder-order transition at several volume fractions, and with particle interacting via both

repulsive and attractive pair potentials.

Presentations

Bolton, C., Debenedetti, P. G., and Russel, W. B., "Dynamics of Disorder-Order Transition in Colloidal Suspensions," Nonequilibrium Particle Aggregation Processes Session, AIChE Annual Meeting, New Yor, 1987.

Determination of Extent of Marangoni Suppression Attainable by Gas Jets in Microgravity

George Washington University
Dr. Robert F. Dressler
Dr. N. S. Sivakumaran
NAG1-325
(NASA Contact: A. Fripp, LaRC)
January 1987 - January 1988

Objectives: The objectives of this research are to: (1) determine by a sequence of numerical solutions which vary the velocity of an argon jet, the maximum reduction in Marangoni convection in a silicon float-zone in microgravity. If the reduction is sufficiently great in the immediate neighborhood of the resolidification interface, this will furnish justification for building the apparatus for use in the Space Station; (2) extend the above program for axisymmetric equations to full 3-D unsteady flow. This will then be used to determine numerically the Critical Marangoni Number M_c for silicon; and (3) if M_c as determined above should be sufficiently low to fall inside the operational range for a μ-g silicon float-zone, then calculations will be made to determine if the argon gas jet can prevent formation of the Marangoni instability.

Research Task Description: A computer program will be developed which will solve the Navier-Stokes equations plus the energy equation to yield the steady-state velocities and other pertinent quantities for Marangoni convection in a μ-g float zone. The equations are in axisymmetric form (2 space variables plus time). Measurements of Marangoni velocities will be made in the experimental setup using an axisymmetric drop of oil in 1-g without a gas jet. When good agreement is shown, then well-posed boundary conditions will be derived for the flow with a heated argon jet blowing tangentially over the free liquid surface. A sequence of such problems will be solved for liquid silicon with varying gas velocity to determine when the maximum reduction in Marangoni velocity will be obtained near the resolidification boundary.

The program will be extended to 3 space coordinates, to be used to calculate when flow instability for silicon will be reached for increasing Marangoni number. Three-dimensional flow will be computed for above-critical Marangoni number with the argon jet blowing, to determine if the unstable flow will be eliminated by the jet.

Progress to Date: We rebuilt the one-g experiment to change from a rectangular container for the liquid oil to an axisymmetric drop, and to redesign the light beam for the new shape. We then took streak photographs with silicone oils of viscosity #50 and #10. Working with Drs. H. Lugt, H. Haussling, and J. Gorski at David Taylor Naval Research Center, we modified and extended their computer program to handle the float-zone problem. Calculations on their slow Apollo computer converged toward steady state too slowly for #50 and #10 oil. We therefore obtained #5 oil from NASA Lewis Research Center, for which our computer program converged. To shorten computing time, we later modified the program to alternate between large and small Δt steps, coupling and decoupling the energy equation. This is necessary because of the very high Prandtl number of #5 silicone oil. Streak photos in the one-g experiment fo the axisymmetric #5 oil drop provided that the numerical solutions were correct. We are now partly

rewriting he Apollo program for compatability on the large IBM 4341 at George Washington University. The first solution will be for liquid silicon using a reverse coupling/decoupling method compared with th oil, because of the very low Prandtl number of silicon. We have also incorporated the revised boundary conditions into the program to describe the action of the heated argon jet.

Publications

Dressler, R. F. and Sivakumaran, N. S., "Non-Contaminating Method to Reduce Marangoni Convection in Microgravity Float Zones," J. Cryst. Growth 88. 148 (1988).

Two Phase Gas-Liquid Flow under Microgravity Conditions

University of Houston
Professor A. E. Dukler

Objectives: The objective of this research is to carry out experiments designed to reveal the mechanisms controlling gas-liquid flow at microgravity and to develop general models which can predict the hydrodynamics and heat transfer under these conditions. This includes predicting flow patterns which exist and their transitions, pressure drop, stability of the flow, the size, velocity and frequency of the long gas bubbles and liquid slugs during slug flow and the interfacial structure during annular flow. The work will also include studies of droplet formation and transfer. This work is to be extended to measurements and modelling of th heat transfer which is controlled by the hydrodynamic behavior.

Research Task Description: Experiments are carried out on the LeRC Learjet. Thirty-six trajectories have been executed with a test loop which consists of a 1.27 cm diameter tube about one meter long. Air and water are metered to the tube and films of the flow are taken through a transparent section using a 400 frames/s camera. In addition, measurements are made of time varying pressure drop, and void fraction. The films are analyzed to give information on the flow pattern, characteristic dimensions of the flow, velocity and sizes of slugs and bubbles, wave frequency etc. Future experiments will involve different fluids, test section sizes and entry configurations. Trajectories provide +/- 0.02 g for 12-20 seconds as indicated by accelerometers.

Heat and Mass Transfer in Zero Gravity

National Bureau of Standards - Boulder Laboratories
Dr. Patricia J. Giarratano
Dr. Vincent D. Arp
Michael Jones
W-16, 170
January 1988

Task A: Transient Heat Transfer Studies in Low Gravity

Objectives: The objective of this work is to provide predictive techniques in the form of computer codes and correlations for applications in the design of heat and mass transfer equipment, especially in systems in which transients occur. Our existing mathematical computer model describes transient heat transfer prior to the onset of gravity-driven fluid motion. The model includes the effect of motion induced by the thermal expansion of the fluid adjacent to a flat geometry heater surface and predicts the temperature profile in the fluid during a transient heat pulse. A near-zero-gravity environment is necessary to study this thermally induced motion because in earth gravity the effect is masked by buoyancy-driven convection in the fluid.

Research Task Description: Mach-Zehnder interferometry was the measuring technique used to study the temperature field in experiments in the laboratory and during two series of flights on the KC-135. Reference (1) contains a description and preliminary zero-g data of this work. This measurement technique proved inadequate for measurement of the temperature fields in the very thin boundary layers developed during the heat pulse. Therefore the research this year has focused on exploring the suitability of a special holographic technique which employs a diffuse light source instead of a collimated beam. Preliminary ground tests using holography has allowed considerable more of the boundary layer to be optically probed.

Progress to Date: Zero-g studies of thermally induced motion in highly compressible fluids have been conducted as part of an ongoing NBS program in heat and mass transfer. This study has employed optical measurement techniques (currently holographic interferometry with diffuse light source) to study the temperature profiles in boundary layers which develop during a transient heat pulse to a flat heater surface and the thermally induced motion which develops from the thermal expansion of the fluid near the heated surface.

The tasks designated for the past year were completed but the last holographic measurements on board the KC-135 were not successful because of experimental difficulties with the exposure of the special plastic film use to record the holograms and with other optical photographic equipment. Therefore, we are requesting funds to repeat the ground and KC-15 measurements in order to complete the work outlined in our previous proposal. This work should substantiate the mathematical computer model which was developed for prediction of transient heat transfer. In addition, the successful application of holographic techniques for temperature density measurements in low-g will provide a powerful tool for future low-g experimental studies which we hope to pursue as outlined in the next task.

Task B. Microgravity Experiments on Heat Transfer to Immiscible Liquid Mixtures with Lower Consolute Points:

Two-phase heat transfer in supercritical fluid (SCF) mixtures in the vicinity of the lower consolute point is important in industrial extraction processes for both product and solvent recovery. Recent experimental work at NBS Boulder has focussed on the measurement of heat transfer coefficients for such mixtures.

Results for the system CO2/n-decane have shown unexpectedly high values when compared with classical film condensation theory. Analogous systems consisting of two immiscible liquids with lower consolute points exhibited similar behavior and, and in addition, revealed the presence of condensate film structures which could be due to the presence of thermocapillary or Marangoni-type flows. Successful modeling of these systems will require the precise nature of the flow to be determined.

Experiments carried out under microgravity conditions would be cleaner in that buoyancy forces could be reduced to a negligible level. Simultaneous measurements of heat transfer coefficients and photographic observation of the condensate film should enable the contribution of the surface-tension driven flow to be established. This would lead to a better understanding of the process occurring in SCF mixtures and to more accurate modeling.

With some modification, experiments could be carried out in the existing apparatus used for the observation of transient heat transfer in KC-135 flights. (Task A above). Both ground-based and flight experiments would be performed. Funding is requested for the coming year to perform the following tasks:

(1) Modification of the existing experimental equipment and instrumentation to accommodate the above heat transfer study;
(2) Ground tests of the equipment and data acquisition procedures;
(3) Zero-g tests on the NASA KC-135;
(4) Theoretical modeling and analysis.

Publications

Giarratano, P. J., Arp, V. D., Owen, R. B., Cezairliyan, A., and Miller, A. P., "Transient Heat Transfer and Thermophysical Properties Measurements in Low Gravity," Adv. Astronaut. Sci., 1987 (in press).

Combined Buoyancy-Thermocapillary Convection: An Experimental Study

Stanford University
Professor George M. Homsy

Objectives Thermocapillary convection is now well-recognized as [illegible]he major sources of convective mixing of fluids in a microgravity er[illegible]nt. It is furthermore apparent that the environment of a spacecraft i[illegible]mpletely free of either residual or transient accelerations that may also cause convection. Thus convection caused by the combined mechanisms of buoyancy and thermocapillary is of interest. In spite of its importance, very few experimental investigations of this class of motions exist, and where they do, the range of parameters studied is often limited. Although the nature of convection is fairly well understood when each mechanism acts separately, very little is known about the structure of the flow and the convective transport in the combined case. Even less is known about possible instability phenomena and instability modes when the relevant parameters become large. Accordingly, we have undertaken an experimental study of the problem in a well-characterized and controlled geometry, namely a rectangular channel.

Research Task Description: We are considering the simple prototype problem of convection in a rectangular channel with a free surface, heated from one side and cooled from the other. The strength and nature of the convection is determined by the aspect ratios of the container, and the magnitudes of the Rayleigh, Ra, Marangoni, Ma, and Prandtl, Pr, numbers. We are generally interested in large Ra and Ma, as convection dominates over conduction in that case. The objectives of the research are to: (1) establish the conditions under which the motion is two-dimensional and steady; (2) characterize the convection by a combination of flow visualization and particle image velocimetry; (3) identify the qualitative nature of the flow as a function of the ratio, Ma/Ra, and (4) observe any instabilities, measure the critical parameters for onset of instability, and characterize them with respect to their temporal and spatial variation. We have chosen a low viscosity silicone oil as the working fluid. Theoretical considerations indicate that the observed phenomena may be most easily interpreted in the case of moderate Pr; our fluid has Pr = 8.5. The apparatus consists of a cell of nominal dimension 1 cm x 1 cm x 4 cm. The combination of fluid properties and physical dimensions allows experiments in which both mechanisms contribute to the observed flows. We employ optical techniques in which a chopped laser sheet is used to illuminate the flow in a vertical plane. The fluid is seeded with small tracer particles, the scattering from which is used for both qualitative visualization and quantitative velocimetry.

Progress to Date: The flow structure observed consists of a relatively large thermocapillary eddy near the free surface, which penetrates deeply into the fluid on the cold wall, but less deeply as the circulation approaches the hot wall. This is attributed to the effects of the stable stratification produced by buoyancy. As Ma and Ra are increased, the flow takes on a boundary layer character, with a strong thermocapillary boundary layer near the free surface, and thin buoyancy layers near the vertical side walls. Thermocapillarity drives a central vortex, while buoyancy produces a stable vertical stratification, limiting the vertical velocity. Serial sectioning of the flow shows that for $Ma<3\times10^{3}$ and $Ra<4\times10^{4}$, the flow is two-dimensional. For parameter values substantially in excess of these, there is strong evidence for a three-dimensional flow pattern which is

spatially periodic in the axial direction. We are currently studying the nature of this pattern, which is indicative of a new instability mode.

Acoustic Forcing of a Liquid Drop

West Virginia University
Dr. M. J. Lyell

Objectives: The objective of this research is to investigate analytically and numerically the acoustic forcing of liquid drops. Specifically, the goal is to develop a formulation in which the tangential radiation stress contributes to forcing of the drop. This will provide for a more complete understanding of the fluid mechanics of forced liquid drops.

Research Task Description: Advanced technology which will enable the manipulation of a liquid sample in a microgravity environment is currently under development. One specific system is the three-axis acoustic levitation chamber which utilizes acoustic radiation pressure forces to not only position but also to manipulate the droplet. For example, the drop can be made to oscillate via forcing at its resonant frequencies. This research investigates the fluid dynamics involved in the acoustic forcing of a liquid drop. In particular, the tangential components of the acoustic radiation pressure vector responsible for tangential forcing will be taken into account. The appropriate governing equations are formulated, and the nature of the coupling between the first order sound field and the second order hydrodynamic field is elucidated. Ultimately, the specific form of the acoustic field and radiation pressure vector are to be displayed. The hydrodynamic field will then be obtained. In the case of non-modulated acoustic forcing, the flow field interior and exterior to the deformed drop will be obtained. This will be done numerically via expansion in spherical harmonics. The response amplitude of the forced oscillating drop will be obtained. Results will be compared to those of experiments. Finally, an extension to the case of acoustic forcing of a fluid compound drop will be done.

Fundamental Study of Nucleate Pool Boiling under Microgravity

University of Michigan
Professor Herman Merte, Jr.
NAG3-663 (NASA Contact: F. Chairamonte, LeRC)
October 1985 - February 1989

Objectives: This research is part of a program for the study of the fundamentals of nucleate pool boiling heat transfer under the microgravity conditions of space, seeking to improve the understanding of the basic processes that constitute boiling by removing the buoyancy effects which mask other phenomena, and which will be part of the development of data base for space applications of boiling.

The initial research focuses on the net forces acting on the growing collapsing vapor bubbles when buoyancy has been drastically reducing, and consist of (a) internal vapor bubble pressure, (b) bulk liquid momentum, (c) molecular momentum of evaporation condensation, (d) liquid viscosity, (e) liquid-solid-vapor surface tension, (f) Marangoni convection, (g) residual buoyancy. It is desired to determine how these forces are related to the temperature distribution in the liquid, which itself is influenced by buoyancy.

Research Task Description: A relatively large (19 mm x 38 mm) flat heat transfer surface is part of one wall of a closed vessel, maintained at a constant pressure and initially uniform temperature. The independent variables are subcooling and heat flux, with a step increase from zero to a prescribed power input. Measurements of surface temperature are made simultaneously with motion photography during the transient heating process, including the onset of boiling, until a terminal condition is reached appropriate to th particular circumstances present. Two heating surfaces are being developed: a semi-transparent layer of gold vacuum deposited on a quartz substrate, which acts simultaneously as a well-defined electrical heater and resistance thermometer, and which permits viewing simultaneously from the side and beneath the boiling surface, and a copper surface indirectly heated electrically, gold coated so as to present the same surface energy conditions to the boiling fluid. Testing will be conducted in the laboratory at $a/g = \pm 1$, and in a drop tower for short term microgravity. Plans are being developed for subsequent orbital flight in the shuttle to provide the longer time periods necessary. R-113 is selected as the initial fluid to be used since it is in a class of "space fluids" such as cryogens, ammonia and fluorocarbons, and being nonpolar is compatible with the use of a transparent gold-film heater carrying an electrical current.

Progress to Date: Analysis of the dynamics of the vapor bubble growth process has begun, seeking to describe the interfacial motion as a result of the various forces. Fabrication of the test package for operation at $a/g = \pm 1$ has been completed, and the various associated electronic control components have been tested and are operational. The gold film heater surfaces have been calibrated, and the metal gold covered heater surfaces fabricated. The system is now ready for use. Fabrication of the test package for operation in the 5 second drop tower at NASA-Lewis is underway. A Science Requirements Document for an orbital experiment is being developed.

Presentations

Merte, H., "Combined Roles of Buoyancy and Orientation in Nucleate Pool Boiling," *ASME Winter Annual Meeting: Significant Questions in Heat Transfer: The Mechanisms of Nucleate Boiling Heat Transfer*, November 1988.

Influence of Time-Dependent Gravitational Acceleration in the Presence of Magnetic Fields on the Fluid Dynamics and Heat Transfer in Solidification Processes

Massachusetts Institute of Technology
Professor S. Motakef
NSG-7645
(NASA Contact: R.K. Crouch, NASA HQ)
October 1, 1986 - September 30, 1988

Objectives: The goal of this research is to determine the effectiveness of various low duration low-g vehicles achieve microgravity conditions during bulk growth of semiconductor crystals.

Research Task Description: This study numerically analyzes the transient response of buoyancy-driven flows on low Prandtl number melts to variations in g-level. Using recorded g-level data from KC-135, space processing applications rockets (SPAR) and TEXUS flights, the results of this analysis are used to establish the duration of low-g periods on board these vehicles.

The transient behavior of natural convection in unidirectional solidification processes has been thoroughly investigated both from fundamental fluid mechanic considerations as well as calculations related to NASA KC-135 aircraft and TEXUS and SPAR sounding rockets. The time constants of convection in the melt of semiconductors to step increases and decreases in g have been calculated in the range of 0-1.5g for the MIT Bridgman-Stockbarger system. The time constant for step increases in g is controlled by the relative dominance of inertial and viscous forces. For step reductions in g, the system response is controlled by the momentum diffusive time scale in the melt Transient analysis has been extended to study the solidification processes on board KC-135 and sounding rockets using the recorded g-level data. The effectiveness of these vehicles is controlled for a given material by the charge size and is, to first order, independent of furnace design. The relationship between the duration of low-g growth vehicles is calculated. The present KC-135 furnaces do not appear to provide the necessary low-g period for meaningful experimentation, whereas TEXUS rocket furnaces do provide sufficiently long low-g growth periods of up to 4 minutes.

The response of convection in the melt to periodic variations in the g-level has also been investigated by conducting a frequency response analysis. The system response is controlled by the momentum diffusive time scale; at oscillating frequencies less than the diffusive time scale, the convection in the melt follows the periodic variations in the g-level, and at higher frequencies the amplitude of the oscillating low velocities decrease linearly with the oscillating frequency. Using the recorded g-level data, it is shown that g-jitter on board KC-135 and sounding rockets, as well as the shuttle, do not significantly interfere with convection in the melt at low-g levels.

Progress to Date: In order to fully account for all transient phenomena during melt growth of semiconductors, we have developed a time-dependent model of the growth process which provides for temporal - tracking of the temperature, velocity, and species concentration fields in addition to the morphology and rate of propagation of the crystal-liquid interface. This formulation allows analysis

of axial as well as radial segregation, consistent calculation of the crystal growth rate (not a priori assumed to be equal to charge translation rate) and study of the effect of the finite axial dimension of the charge. Over the past six months we have essentially completed the main elements of the numerical code and presently performing bench-mark studies.

Publications

Griffin, P. R. and Motakef, S., "Analysis of Effectiveness of Low-Duration Low-Gravity Vehicles during Unidirectional Solidification of Semiconductors," J. Cryst. Growth, 1988 (in press).

Energy Stability of Thermocapillary Convection in Models of the Float-Zone Process

Arizona State University
Professor G. Paul Neitzel
Professor Daniel F. Jankowski
NAG3-568
(NASA Contact: Dr. A.T. Chai, LeRC)
September 1987 - September 1988

Objectives: The objectives of this research are to calculate energy-stability limits for basic states which are models of the float-zone crystal growth process. Energy-stability limits provide sufficient conditions for stability of a basic state. The successful computation of such a limit for an actual float-zone basic state would identify conditions under which oscillatory convection (and hence striations in the final material) would be absent.

Research Task Description: Finite-element and finite-difference methods have been used to calculate the basic state in a model of the float-zone, crystal-growth process. A temperature gradient along a nondeformable free-surface generates thermocapillary convection. The energy-stability limit (Ma_E, a value of the Marangoni number below which stability is guaranteed) for this basic state has been calculated from discrete versions of the quadratic functional that defines the limit. The numerical procedures have been verified by their application to the basic state in a cylinder heated from below.

The problem has provided to not only a significant undertaking from the standpoint of fluid mechanics and stability theory, but also requires state-of-the-art computational techniques and computer resources. In particular, the stability calculation requires the treatment of large, sparse, generalized, algebraic eigenvalue problems without the property of positive-definite matrices. In addition, the problem has many parameters which hinder an orderly presentation of the results. However, for a certain meaningful range of these parameters, there is reasonable agreement between the finite-difference and finite-element stability results. Both methods show the expected increase in Ma_E with increasing buoyancy. Both methods show that the "linking parameter" can have a significant effect on the stability results, with Ma_E increasing as this parameter decreases. In spite of these positive comparisons, there is a difficulty associated with varying Prandtl number that is currently being investigated.

Progress to Date: Progress is also being made on the treatment of the problem with a deformable liquid-gas interface. A code capable of calculating the basic state is being de-bugged and the formulation of the stability problem for this basic state is underway.

Publications:

Neitzel, G. P., Shen, Y., and Jankowski, D. F., "Energy Stability of Thermocapillary Convection in a Model Half-Zone, Finite-Difference Results," Abstract, *40th Meeting of the Division of Fluid Dynamics, American Physical Society*, November 1987.

In-Space Experiment on Thermoacoustic Convection Heat Transfer Phenomenon - Experiment Definition

University of Tennessee, Knoxville
Professor M. Parang

Objectives: The objective of this research is to define a potential flight experiment to evaluate the significance, enhance the understanding and to access the phenomenon of thermoacoustic convection and its effects on fluid storage handling and transport, as well as, material processing in space environment.

Research Task Description: The research tasks include: (1) conduct numerical and supporting ground-based experiments to help establish and define the objectives of the in-space experiments and the necessary technical requirements for the experiment hardware; (2) develop conceptual designs for the experiment; and (3) develop preliminary plans, schedules, and costs for implementing the experiment aboard a shuttle mission.

Progress to Date: The funding for this research began on August 1, 1988. Progress to date includes numerical modeling and computation of the thermoacoustic effects in an enclosed fluid. Also preliminary considerations for the design of ground-based experiments are in progress.

Breakdown of the Non-Slip Condition in Low-Gravity

Los Alamos National Laboratory
Dr. Donald R. Pettit
C-32005-K (NASA Contact: R.A. Wilkinson, LeRC)
May 1988 - May 1989

Objectives: The objective of this research is to investigate anomalous slip of liquids at a solid wall seen previously in low gravity.

Research Task Description: The purpose of the experiment is measure the extent of fluid-wall slip in a concentric cylindrical cell where the fluid fills the annular gap. Couette flow is introduced into the fluid by rotating the inner cylinder at constant RPM. Alteration of the boundary condition is determined from the torque ratio between low-g and normal g. The torque ratios previously measured have been between 0.96 and 0.91, showing a decrease in torque during low gravity.

Progress to Date: Funding for continuing this task was not received until July 1988. As of this date, some new equipment has been ordered but not received. When the equipment arrives, the hardware will be assembled and flown sometime late fall or winter depending on the availability of KC-135 flights.

Fluid Dynamics and Low Gravity Effects of Chemical Vapor Transport

University of Alabama in Huntsville,
Center for Microgravity and Materials Research

Dr. Franz Rosenberger
Dr. Thomas Nyce

Objectives: Chemical vapor deposition (CVD) processes are used on a large scale for the preparation of electrically active and insulating layers for opto-electronic solid state devices. The performance of these devices often depends sensitively on the uniformity in thickness and composition of the as-grown layers. These, in turn, depend upon the heat and mass transfer conditions during the deposition process, i.e. on the fluid dynamics in the nutrient vapor phase. Currently, the fluid dynamics of CVD processes is not too well understood, though recent research has revealed that buoyancy-driven convection is responsible for various layer non-uniformities, that seriously limit the yield of CVD materials processing.

This research program will delineate the effects of gravity in current CVD practice, give guidance in reactor design for reducing these effects on earth, and explore the potential of CVD experiments under low gravity conditions. Beyond these questions of great technological importance, this research can be expected to clarify some fundamental aspects of combined (superimposed buoyancy-driven and forced) convection.

Research Task Description: A complimentary theoretical and experimental research program is being pursued to quantify the effects of gravity ($1\text{-}10^{-6}$g) and total pressure (760-0.1 torr) on compositional and thickness uniformity attainable in horizontal reactor geometries used in silicon CVD and GaAs MOCVD. The work includes extensive numerical modelling (3-D, compressible, multicomponent steady-state flow, including homogeneous gas phase reactions and some account of interfacial kinetics). In the experimental part of the study, velocity distributions in CVD reactor models are being determined with laser Doppler anemometry, with some emphasis on the characterization of the parameter combinations that lead to (undesirable) non-steady transport.

Progress to Date: In order to clarify the limitations of 2-D theoretical modelling and to establish a well-defined foundation for our 3-D modelling, we have performed a systematic numerical study and compared the results with published experimental data for the MOCVD of gallium arsenide. This study shows that 2-D models can produce realistic results for the growth rate distribution only for reactors with large cross-sectional width-to-height (aspect) ratios that are operated at subcritical Rayleigh numbers. In addition, Soret diffusion has been identified as a major factor for growth rate uniformity. Furthermore, velocity corrections for finite aspect ratios must be made, and buoyancy effects can be significant even in large aspect ratio reactors at the leading edge of the susceptor. Whereas the temperature dependence of the transport parameters can be ignored for the prediction of growth rates (due to a compensating effect of property averaging on the diffusive and convective fluxes at the susceptor), the composition dependence of the transport properties must be retained in a realistic model

The experimental studies, conducted on a model reactor with aspect ratio 2, have shown that, depending on the underlying Reynolds and Rayleigh numbers, combined convective flows cover three distinctly different flow regimes: (a) Steady flow

throughout the entire channel with two maxima in the crosswise profile of the axial velocity developing symmetrically about the vertical center-plane; (b) Steady flow throughout with asymmetries about the center plane; and (c) Non-steady flow with asymmetries about the center plane evolving with increasing distance from the leading edge of the heated susceptor for Rayleigh numbers in excess of 14,000. The finding of these unexpected asymmetries, is extremely important for numerical modelling. All CVD modelling work to-date has assumed (computer time-saving) symmetry of the flow about the vertical center plane.

Publications

Ouazzani, J., Chiu, K-C., and Rosenberger, F., "On the 2D Modelling of Horizontal CVD Reactors and its Limitation", J. Cryst. Growth 91, 497-508 (1988).

Influence of Hydrodynamics on Capillary Containment of Liquids in a Microgravity Environment

Cornell University
Dr. P. H. Steen
NAG3-801
(NASA Contact: Dr. S.A. Kassemi, LeRC)
June 1, 1987 - May 31, 1989

Objectives: The objective of this research is to determine under what conditions in a low-gravity environment small-to-moderate shear stresses inhibit the capillary instability of a liquid bridge. Hydrodynamic stability theory is used to predict windows in parameter space where stabilization occurs in model systems and these predictions guide ground-based experiments which utilize a Plateau-chamber. This apparatus simulates low-gravity by matching the densities of two immiscible liquids.

Research Task Description: The microgravity environment of space allows the containment of liquids by surface tension in a range of configurations not possible in conventional ground-based applications. The distortion of the free liquid interface by gravity may be suppressed to advantage in containerless energy exchange, in the transportation of liquids, and in the processing of materials, to name a few.

The molten metal region of the float-zone refining process is a well known example where containerless confinement is exploited; the small gravity force relative to surface tension allows a somewhat longer float-zone configuration than otherwise possible. Nevertheless, this feature of low gravity has not been emphasized largely because the enhancement of zone-length is limited by the surface-tension instability. Even in zero-gravity, surface tension constrains a circular cylinder of liquid to be shorter than its circumference; longer cylinders tend to break up into droplets. Indeed, wherever surface tension is used to advantage, the shape of the contained liquid is limited by the capillary instability; only shapes of small aspect ratio are possible. Although most modifications of a dominant capillary force by other forces destabilize the configuration, both experimental and theoretical evidence suggest that certain shear stresses and pressure distributions induced at an interface can stabilize the capillary break-up. The ability to modify the geometry of free liquids to include long continuous configurations (large length to diameter) has far-reaching and broad technological implications for space application.

Progress to Date: Based on the analysis of a cylindrical interface which, by means of surface tension, contains a viscous liquid set into axial motion, we have found conditions for which the capillary break-up is completely stabilized.

The annulus of liquid includes geometries which vary from a jet with a wire down the center to a thin film on a large drum. We find that complete stabilization is achieved (all wavenumbers decay) for thin layers flowing along a rod driven by shear forces which are moderately but not too large.

In the experiments axial shear stresses are imposed at the interface between the bridge and the immiscible, neutrally buoyant surrounding liquid by two alternative means: (i) a thermocapillary induced surface-tension gradient, or

(ii) a (isothermal) pipe flow of the outer fluid. Experiments using method (i) may be viewed as a model of thermocapillary convection in a float-zone process. Auxiliary experiments which involve a soap-film bridge across the fundamental question of stabilization, their advantage is simplicity. Experiments are in progress.

Publications

Russo, M. J. and Steen, P. H., "Shear-stabilization of the Capillary Break-up of a Cylindrical Liquid Annulus."

Presentations

Russo, M. J. and Steen, P. H., "Instability of Thermocapillary Driven Annular Flow," Division of Fluid Dynamics, American Physical Society Annual Meeting, Eugene, OR, November 22-24, 1987.

Russo, M. J. and Steen, P. H., "Shear-stabilization of Capillary Break-up," Division of Fluid Dynamics, American Physical Society Annual Meeting, Buffalo, NY, November 20-22, 1988.

Cryer, S. A. and Steen, P. H., "Rupture of a Soap-film Bridge: Stability to Large Disturbances," AIChE Annual Meeting, Washington, DC, November 26-December 2, 1988.

Ostwald Ripening of Solid-Liquid Mixtures

Northwestern Reserve University
Dr. P. W. Voorhees
S. C. Hardy, NIST
H-85025B (NASA Contact: D. Frazier, MSFC)

Objectives: The objective of this program is to use the unique conditions provided by space flight to study the kinetics of Ostwald ripening. The data derived from this experimental work will provide baseline data for the field and thus permit the refinement of existing theories of the kinetics of first-order phase transformations. In addition, as the Ostwald ripening process has a major impact on the properties of materials, the experimental results will yield information which can be used to improve the properties of materials containing dispersed phases.

Research Task Description: A particularly ideal system to use in these experiments is a mixture consisting of solid particles in a liquid. Since the coarsening rate in such a system is comparatively fast, and in a properly chosen system the solid particles can be spherical, the experiments can serve as a careful test of theory. However, experiments performed using a low volume fraction solid, where the theory is most accurate under terrestrial conditions, shows that buoyancy driven convection of the solid particles is prevalent and thus the experiments do not satisfy the theoretical requirements of fixed spatial locations of the particles. To eliminate this problem the experiments will be performed in the reduced gravity environment of space.

Previous Work: We have located a solid-liquid mixture in which the material parameters required to compare the experimental results to the theoretical predictions are known and have developed an experimental procedure necessary to produce a dispersion of solid particles in a liquid. We have examined the coarsening kinetics of solid particles in a liquid for the volume fraction solid range about 0.4 where the development of a solid skeletal structure inhibits particle sedimentation. The experimentally measured coarsening rate constants are found to exceed those calculated from theory by factors ranging from 2 to 5. Possible causes for the disagreement between theory and experiment are the movement of particles within the skeletal structure due to density differences between the solid and liquid phases, coalescence of the solid matter, and convection. Experiments in a microgravity environment will eliminate these two possibilities.

Another possible cause for the disagreement between theory and experiment is an enhancement in the ripening kinetics due to the transport of latent heat. Thus, we have investigated the role of ripening in which systems which specifically account for the transport of heat, latent heat and mass. This theory shows that the ripening kinetics with only mass addition to the system kinetics can be significantly faster than the predictions of a theory which neglects the effects of the flow of latent heat. However, the theory also shows that the undercooling in the systems employed in the experiments are sufficiently high that the transport of latent heat has a negligible effect on the ripening kinetics and cannot account for the disagreement between theory and experiment.

Finally, experiments have been initiated to investigate the effects of imposed fluid convection on the ripening process. In this case, solid-liquid mixtures of lead-tin are rotated, with the axis of rotation perpendicular to the gravity vector. Preliminary experimental results show that even though a stable skeletal structure is present, the slow rotation rates employed in these experiments can cause an enhancement of the ripening kinetics and, in some cases, a change in the exponent of the temporal power law for the average particle radius from the classically predicted value of 1/3. Further investigation of the effects of rotation are underway.

Publications

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Collision and Coalescence Studies

Jet Propulsion Laboratory
Dr. Taylor G. Wang
In-House

The objective of this investigation is to study three aspects of the collision and coalescence of uncharged free drops: time dependent deformation of a drop upon collision, dynamics of air-film drainage, and drop stability after coalescence.

Spacelab provides a unique environment of near-weightlessness for conducting science in space, and thus will offer a unique opportunity to obtain quantitative data on the dynamics of collision and coalescence of liquid drops. The proposed experiments will utilize a modified version of the Drop Physics Module (DPM) provided by NASA to obtain accurate quantitative data on the behavior of collision and coalescence of liquid drops in microgravity conditions. These investigations will be free from the constraints imposed by the influence of Earth's gravity (including insufficient droplet sizes for accurate observation), and will extend for periods of time unattainable even in the longest rocket flight tests.

The results of the investigations will be used to verify existing linear theory, and to provide the necessary insight for further theoretical development of the subject. The deficiencies of the existing theory, which disregards nonuniformity of the air-film drainage, irregularity in the surface of separation, rotational energy, and oscillation energy, are exemplified by inconsistent results appearing in the literature.

In 1988 we have performed preliminary collision and coalescence experiments in an immisible system with acoustic as the driver force. The static deformation of two impinging drops compare favorably with the calculation, but the final rupture shows a resonance effect. Whether this is an art effect due to the experimental set up or something significant will be investigated in the following year.

C-2

Transport Processes in Solution Crystal Growth

NASA Lewis Research Center
Dr. R. A. Wilkinson
In-House

Objectives: The objective of this task is to conduct fundamental research on non-intrusive direct measurement techniques useful in quantifying transport properties of solution crystal growth systems. The utility of this effort is to enable study of diffusion limited transport in low gravity solution crystal growth.

Research Task Description: This activity is being conducted to evaluate the science feasibility of utilizing Raman spectral scattering signals to provide improved spatial resolution measurements of concentration profiles near a crystal interface during growth or dissolution of KH_2PO_4 (KDP). Optical multichannel detection of a solute vibrational band provides direct quantification of solute concentration with band intensity. Optical fiber sampling of incident laser intensity provides a synchronized multichannel record for data quality control. Crystal growth is induced by cooling the crystal substrate such that the solution near the crystal surface is supersaturated. The Raman band intensity is not sensitive to the few degree centigrade temperature variations induced. The spectroscopic sample volume is documented with optical microscopy.

Progress to Date: Precision calibration of Raman intensity vs. KDP concentration at sub 1.0% standard deviation error levels has been demonstrated over a worst case spread of optical set-ups and time drift of instrumentation. A fiber optic, sampling incident laser intensity, piping light to unused optical multichannel analyzer (OMA) channels, had to be implemented to guarantee data quality. It provided a fully synchronized monitor of fluctuations in laser power to correlate with observed Raman signals. With 1 W of laser power at the sample (transparent) and a 50% efficient throughput spectrometer in the detection path good data statistics required 8 repeated data sets at approximately 2.5 minutes per set. The roughly 20 minutes accumulated represents the time to measure concentration at one spatial location. 35mm photomicroscopy was implemented to document the 30 micron dia. by 200 micron long laser Raman scattering region in the solution with respect to the crystal surface. The laser beam was able to approach up to 25 microns from the crystal surface. During solution crystal growth scattering of nucleated microcrystals in solution caused some intensity noise. These microcrystals convect right up to the crystal surface indicating no quiet diffusion region under normal gravity conditions. Solute Raman intensity reflected from the crystal surface in some configurations was weakly detectable. Focal length changes resulting in optical misalignment and signal loss due to sample translations are characterized.

The work has concluded at this point. A NASA TP has been written and is in the Report Control system. The intent of the TP is to give crystal growth practitioners the substance to evaluate this technique and justify further activity if merited.

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Molecular Dynamics Fluid Simulation

NASA Lewis Research Center
Dr. R. A. Wilkinson
In-House

Objectives: The objective is to establish a molecular level equilibrium and non-equilibrium fluid modeling capability simulating presence or absence of gravity and its effects on transport and surface phenomena. Molecular Dynamics (MD) simulation provides a means to get low gravity information on fluid behavior that can complement and optimize microgravity in-space and ground based experiments. The activity will develop the algorithm and apply it to a variety of problems; for example, surface tension, surface configuration, and momentum transport near a wall.

Research Task Description: The activity entails, in addition to developing the main program that numerically integrates molecular equations of motion, developing algorithms to compute continuum fluid local density, velocity, pressure sensor, structure factor, diffusion coefficient, heat flux, surface tension, and surface excess quantities. The first task to be pursued will be the characterization of shear momentum transport with and without gravity near several models of a solid wall. An important check point will be to examine at what level of computer precision will gravity effects be detectable on particle positions and velocities and the averaged continuum properties. To support these calculations PC based dedicated parallel processing hardware is to be setup. A PC will be the front end and possible graphics processor for a Transputer network. It will be configurable and scalable to any MD problem and allow dedicated full time processing. A Parallel construct FORTRAN 77 compiler designed specifically for Transputer hardware will provide the software environment with some code possibly programmed in the Transputer's native OCCAM.

Progress to Date: A desktop PC based MD algorithm has been developed for molecules in the liquid state flowing near a non-wetting crystalline and idealized smooth walls. A Lennard-Jones potential with xenon parameters is being used to model the liquid. Several possible tests for equilibrium or sded for 12 picoseconds. The code developed is transportable from a PC, for small number systems, to the Cray, for large systems. However, the Transputer hardware platform is now the preferred alternative. A set of continuum fluid properties can be calculated from the main algorithm output. To that end supporting algorithms have been added.

While the desktop PC has continued to equilibrate the system, some Transputers have been procured and set up. Learning OCCAM and parallel programming concepts and techniques continues.

4. BIOTECHNOLOGY

Protein Crystal Growth in Low Gravity

Center for Materials Research, Stanford University
Professor Robert S. Feigelson
Dr. Robert C. DeMattei
NAG8-489
(NASA Contact: M. Pusey, MSFC)

Objectives: The ultimate objective of this research is the careful design of an experiment to study the effect of low gravity on the growth of protein crystals in a long-duration space flight. The proper design of the flight hardware and experimental protocols are highly dependent on understanding the factors which influence the nucleation and growth of crystals of biological macromolecules. Thus, the primary objective of this research is centered on investigating these factors and relating them to the body of knowledge which has been built up for "small molecule" crystallization. This data also provides a basis of comparison for the results obtained from low-g experiments.

Research Task Description: The main component of this research program is the study of mechanisms of protein crystal growth and those parameters which influence growth. Canavalin was chosen as a model protein to use in the studies. The program resolved itself into six tasks:

1) Solubility Studies - This research was designed to determine the solubility of canavalin and its dependence on temperature and solution parameters.

2) Growth Rate Studies - By studying the growth rate dependence of canavalin on supersaturation, the rate determining step of the growth process can be inferred.

3) Localized Supersaturation Control - Using the data from the solubility studies, methods of controlling local supersaturation in order to control nucleation events can be investigated.

4) Flow Imaging Studies - a study of the applicability of Schlieren imaging techniques to flows developed during protein crystal growth. In addition to canavalin, lysozyme and Rochelle salt will be used as models.

5) Protein Growth Model Development - Data gathered in this and other research will be used to develop a predictive model for protein crystal growth.

6) Space Flight Experiment - a conceptual design for a long-term space flight experiment eill be developed.

Progress to Date: The solubility of canavalin was determined. Its dependence on pH and temperature was srudies and it was determined that cabavalin behaves in a "normal" manner, i.e., it is less soluble at lower pH (high hydrogen in concentration) and lower temperature. An estimate of the heat of crytallization was made based on solubility data.

The dependence of growth rate of canavalin crytsals on supersaturation was studied at 20°C. The fitting of the data to various growth models determined that the rate limiting step in the growth of canavalin was a screw dislocation interface attachment

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Cell Partition in Aqueous Phase Systems

University of Alabama in Huntsville
Dr. J. Milton Harris
NAS8-35362
(NASA Contact: R.S. Snyder, MSFC)
July 1987 - July 1990

Objectives: The goals of this work are to understand and control low-g demixing of two-phase systems formed by solution of pairs of polymers (generally dextran and polyethylene glycol) in water and then to use these systems to purify cells by partitioning. The immediate objective has been to control demixing by controlling wall wetting with covalently-bound polymer coatings.

Progress to Date: Ground-based experiments show that we can achieve esentially any contract angle between the phase interface and the container wall by using dextran coatings of different molecular weights. Our recent experiment on STS-26 showed that we can control localization of the phases. Next we will focus on controlling the rate of remixing and on performing cell separations in space.

Publications

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Separation of Chromosome-Size DNA Molecules

University of Pennsylvania
Professor Ponzy Lu
Young Cho, Drexel University
Lee Silver, Princeton University

Objectives: This project addresses the problem of DNA separation by gel electrophoresis. Since we are interested in DNA molecules 10^6-10^8 base pairs in length (10^9-10^{11} daltons), or molecules that are polyanions of 2 nm diameter with lengths of millimeters to many centimeters which distort as the move through the electrophoretic matrix. Existing theories and models for electrophoretic transport processes must be modified. The goals of the project are:

1) Directly observe the orientation of the DNA molecules in the electrophoretic process in the gel matrix
2) Directly measure the motion of the DNA chain constrained in the gel and its reorientation as the applied electric field is pulsed.
3) Use the results from the direct observations to evaluate and modify existing theories and models for DNA electrophoresis.
4) Explore the use of alternates to agarose to seek rigid media with greater porosity to extend the current 10Mb upper bound in pulsed field DNA separation methods.
5) Explore the use of magnetic field to influence the shape and tertiary structure of the DNA to scale the DNA dimensions so that existing electrophoresis media can be used with longer DNA molecules.
6) Explore the use of natural DNA packing proteins to scale the DNA dimensions so that existing electrophoretic or gel permeation media can be used for their separation.
7) Identify those separation parameters that can be enhanced by a microgravity environment.

Research Task Description: This effort will complement parallel national efforts to map and sequence the entire human genome. There are currently limitations in the human genome project which can be addressed by the technologies to be developed by this research project. Even if the human genome project is completed using existing methods, the technology to be developed here will extend the benefits of those efforts to all biological species, including those used as models of human disease and for agriculture and industry.

This project is an extension of NASA support of electrophoretic separation process development over the past decade. The extrapolation of currently employed methods for DNA separation suggests the use of more dilute electrophoretic media to allow larger porosity and consequently shorter reorientation times of the polymer subjected to pulsed electric fields. The only method to maintain fluid stability in the presence of chemical and physical density heterogeneity is the microgravity environment. Since the separated DNA will be used for genetic analysis, this program will keep space science applications at the cutting edge of biotechnology.

Progress to Date: Project officially began September 1, 1988. First purchase orders were allows to be placed October 15. Progress to October 31 includes the appointment of 2 postdoctoral fellows and one graduate student. Scientific progress has been the discovery that bent copperwire dropped in Newtonian and non-Newtonian fluids may be a useful mechanical model of DNA gel electrophoresis.

Biological Separations in Space

Johnson Space Center
Dr. Dennis R. Morrison
In-Center
January 1985 - present

Objectives: This research includes ground-based research and analysis of two flight experiments using Continuous Flow Electrophoresis (CFE) to separate living cells under microgravity conditions. It involves comparisons of various commercial devices to purify target mammalian cells, hormones, enzymes, and development of micro-analytical assays for secreted cell products. The research is conducted in collaboration with Baylor College of Medicine, The University of Texas Health Sciences Center, Penn State University, and the National Institute for Standards and Technology at Boulder, Colorado.

GROUND CONTROL RESEARCH

Ground-support research includes: 1) comparative separations with commercially available CFE devices and other free-fluid electrokinetic separation methods; 2) studies of specific cell-cell interactions which may be altered in microgravity; and 3) collaborations with academic institutions involving purification of secreted cell products and development of ultra-sensitive product assays required to properly interpret the microgravity experiments. Results include new ELISA and chromogenic assays of secreted cell products, measurements of target cells using fluorescence and flow cytometry, and a new cell system for studies of immune cell function in microgravity.

1. Comparative Electrokinetic Separations

The limitations on ground-based systems prohibit separation of enough target cells per fraction at the required resolution to characterize the unique relationships between secretory function and their net surface charge. However, comparisons of EPM distributions from actual separations on the ACE 710 (Hirschmann) and CFES (McDonnell Douglas Astronautics Co.) have provided new insight into operational variables which can be improved in conventional systems. Although the residence time and throughput are more limited in the ACE 710, direct comparisons with the CFES have revealed that the ACE 710 can serve as a valuable tool in development of flight separations on the CFES. The ACE 710 is designed to operate with much higher conductivity buffers (> 600 uS) thus cooling limitations restrict the field strength to less than 100 v/cm and residence time to approximately 30 seconds. The relative separation potential (Et) can be estimated from the product of the field strength (v/cm) x residence time in minutes. The maximum practical Et for the ACE 710 is approximately 50 compared to a demonstrated Et of 300 for the CFES. In separations of the same cell lots the effective resolution of the ACE 710 does not show the degree of electrophoretic heterogeneity which characterizes rat pituitary cells or mouse lymphocytes. However, the ACE 710 data (Et=50) can be used to predict the bandspread of CFES fractions that will be collected from the CFES at a higher separation potential (Et=150).

2. Development of a Cell System to Study Specific Lymphocyte Functions

Non-immune SJL spleen cells were fused with Balb/c 653-myeloma cells to form hybrid B-cell clones which were tested for antigen presentation capability to T-cell lines that were specific for either lysozyme or myoglobin. Approximately 50% of the B-cell clones were able to present both myoglobin and lysozyme while the other 50% of the clones

presented specifically either myoglobin or lysozyme (specific presenters). This indicates that MHC restricted antigen presentation at the clonal level can be specific or non-specific depending on the particular B-cell clone. Previous studies have used antigen specific T-cell hybridomas to study mechanisms of antigen presentation by B cells which required accessory factors i.e., IL-1. The system we have developed is less complex, enabling experiments to focus directly on B cell antigen presentation to antigen-specific T cells. This cell system is being used to develop new experiments to study lymphocyte functions in microgravity.

3. Urokinase Assays

Urokinase and other plasminogen activators activate the body's fibrinolysis mechanism which dissolves blood clots after they are formed. We have shown that cultured subpopulations of human kidney cells separated by CFES will produce two different molecular forms of urokinase (uPA); single-chain proenzyme (scuPA); PA inhibitors; and tissue-plasminogen activator (t-PA). The bioassay for uPA now depends on measurement of enzyme activity against fibrin or an artificial substrate. More sensitive methods are needed to measure uPA which is secreted by cultured cells. Professor Atassi at the Baylor College of Medicine has developed synthetic peptides analogous to selected regions of the urokinase molecule. Polyclonal antibodies are being developed using the custom peptides as antigens. Cessation of NASA funding has prohibited the development of specific monoclonal antibodies, however, comparisons will be made between the S-244 chromogenic assay and measurements using the polyclonal antibodies in an ELISA assay which could be used in future Shuttle missions.

ANALYSIS OF KIDNEY CELLS SEPARATED IN MICROGRAVITY

Continuous Flow Electrophoresis (CFES) experiments on STS-8 separated human kidney cells into more than 33 subpopulations which were cultured for periods of up to 24 days. The mean mobility for the flight cells was approximately 30 percent greater than the ground controls. The breath and heterogeneity of the EPM distribution for the space experiments was also increased. Calculations indicate that six to eight of the lowest mobility fractions were not collected in the flight experiments. Four, eight and twelve day cultures have been tested for production of urokinase (uPA) and tissue plasminogen activators (t-PA). High production levels were observed in approximately 30% of the cell fractions. Fractions producing high levels of uPA were different from those which produced high levels of t-PA. Comparisons of different assays on the same samples showed that some fractions also produced uPA inhibitors or single chain urokinase (scuPA) which later degraded into the active two chain forms. Ultrasensitive assays are still needed to precisely determine the levels of different forms of urokinase secreted into culture medium by the flight cells.

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Cell Maintenance Systems and Inflight Sample Handling

NASA Johnson Space Center
Dr. Dennis R. Morrison
In-Center
April 1985 - continuing task

Mammalian cells are difficult to maintain alive outside of an incubator for more than a few hours. Cells require precise control of temperature, pH, dissolved oxygen, and pressure in the immediate environment. These requirements must be considered when cells are taken into space. Methods are being investigated to measure cell tolerance to less than optimum environment conditions and to develop appropriate small scale hardware and techniques for maintenance culture, harvesting, fixation and storage of living cell specimens used in microgravity bioprocessing research.

Studies have demonstrated that a specific group of "stress response proteins" are synthesized by mammalian cells in response to temperature stress, dissolved oxygen deprivation, and subnormal glucose levels. Experiments have been conducted using 2D-PAGE to determine the patterns of intracellular protein synthesis for human embryonic kidney (HEK) cells exposed to three different experimental conditions: (1) normal growth conditions in quiescent medium (37°C), (2) two hours at 42 C (temperature stress), and (3) exposure to shear stress (12 dynes per square centimeter) in a laminar flow chamber for two hours. The patterns of intracellular protein synthesis were very different under each of these three conditions. For temperature-stressed cells, at least eleven intracellular proteins were observed in 2D-PAGE gels that were not evident in the control cells. For the shear-stressed cells, at least fifteen new intracellular proteins were observed that were not evident in the control cells. A commercial Fluoromeasure device has been evaluated for application in measuring cell metabolism during shear-stress studies on orbit.

New multi-compartment syringe devices are being developed for experiments which require mixing of living cells with regulating hormones or other cells after reaching orbit. The Cell Transport Assemble (CTA) flown on STS-8 is being redesigned to maintain live cells longer than 48 hours. Small scale apparatus are being developed for harvesting cells and concentrating the cell suspension, and removing cell-free medium for inflight assays. Sterile containers, transfer apparatus and procedures are being developed in conjunction with an advanced CTA which can be used to support cells separation and cell biology experiments.

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Research in Biological Separations and Cell Culture

University of Texas Health Sciences Center
Dr. L. Scott Rodkey
NAS9-17403
(NASA Contact: C. Sams, JSC)
January 1988 - January 1989

The Bioprocessing Research Center (BPRC) underwent significant changes in direction in Year Three of the contract. Dr. Rodkey initiated a series of changes and chose to pursue may of the cell separation projects that were ongoing at the time of his appointment as Director of BPRC. Some experiments were initiated in an effort to study the nonreproducibility of cell separation that had been attempted to that time. Data was collected which clearly showed that the lack of precision and resolution in cell separations is a direct function of the inappropriate buffers that has been chosen to be used in the McDonnell Douglas continuous flow electrophoresis system by early investigators. The nonreproducibility and poor resolution of cell separation systems has been solved. The correct buffer, is, at present, unknown. However, it is now quite clear that the next logical effort in this area of cell separations by continuous flow electrophoresis needs to be in finding an appropriate buffer system which buffers at the pH at which the system operates.

Studies of recycling isoelectric focusing, progressed well during the year. Studies were completed in comparison of the McDonnell Douglas continuous flow electrophoresis system with recycling isoelectric focusing system and the results are unequivocal. During the year, ampholyte synthesis was refined and extended so that new classes of ampholytes are now available for high resolution work. Studies of electrodissociation were carried out, as were studies of ampholyte-protein interactions. Finally, a new design of recycling isoelectric focusing hardware was built and tested during the year.

Another area that has been pursued this year has been that of studies of electrophoretic properties of sickle cells. These studies are ongoing. A new set of studies have been initiated in which precancerous liver cells from rats kept on a liver cancer producing regimen of diet have been studied. These cells are quite heterogeneous and studies indicate that some separation has been achieved.

The following tasks were carried out by colleagues at the University of Houston and Rice University.

Classical industrial and research methods of growing human cells have been limited to systems which grow and maintain cells in a monolayer. This is largely due to the anchorage dependence of mammalian cells. Virtually all normal human cells must be firmly attached to some suitable surface before they will grow, multiply, and eventually secrete with monolayer cultures. The primary goal of this work was to study the fluid dynamic phenomena in bioreactors currently used for mammalian cell culture. This includes the identification of important fluid mechanical quantities in bioreactors and the development of means to obtain information about these quantities. To study the fluid mechanic environment of bioreactors an experimental approach was taken. The size of 500 ml of the bioreactors under consideration made experimental studies attractive, since 1:1 scale models of bioreactors can be used. Thus, problems of transferring results in the proper scale could be avoided. The hydrodynamic environment in some current bioreactor designs have been studied. All designs are perfusion-type reactors with a cylindrical vessel of 500ml and a spin filter for removing

products and waste. For mammalian cell culture, the reactor vessel is designed to grow cells on microcarriers. The designs differed in their agitation schemes. For experimental investigations, several flow visualization techniques were applied to obtain the general flow pattern and qualitative information about fluidization and mixing. Point-velocity measurements were conducted with a laser Doppler velocimeter (LDV). Velocity profiles and estimates of wall shear stresses were deduced from the point velocity measurements. Fluidization, transport/mixing, and low shear stresses were identified as important considerations for bioreactor designs.

Dr. Charles Goochee, of the University of Houston, conducted research in the development of methodology for measuring the stress level of cells in culture, and in the development of a deeper understanding of how cells cope with environmental stress. He is currently developing techniques for quantifying the synthesis rates and concentrations of the intracellular proteins which are most sensitive to environmental stress. Ultimately, it will be possible to discern the specific source(s) of stress for mammalian cells in any bioreactor configuration based on analysis of the profile of intracellular protein synthesis. Two-dimensional polyacrylamide gel electrophoresis combined with protein labeling using radiolabelled amino acids provides a powerful technique for monitoring the cellular response to its environment. It is possible to compare the overall rate of protein synthesis between experimental and control, and to compare the relative rates of synthesis and intracellular concentrations of the 1000 most abundant intracellular proteins. It is possible that these techniques could provide a fairly rapid mechanism for screening the response of various types of mammalian cells to hypergravity. Cells which are found to have altered rates of protein synthesis or altered patterns o intracellular protein synthesis would represent particularly good candidates for further experiments.

Dr. E.T. Papoutsakis of Rice University conducted research in the fluid mechanics and mixing of cell culture bioreactors. In this research the PI is attempting to define the effect of agitation on the growth of tissue cells on microcarriers or freely suspended. Experimental work has been underway since 1986 in two bioreactors to confirm some of the predictions. The emphasis is on the design and operation of the reactor system, rather than on the biology of how the cell responds. The latest results showed that the reduced growth rate of cells was a result of both cell death as well as reduction of the intrinsic cell growth rate. The negative effect of agitation on cell growth was due primarily to the interaction of microcarriers with the smallest eddies of turbulence as well as interactions among specific, quantitative expressions for these interactions had been developed and experimentally confirmed. The PI has carried out a few preliminary experiments and designed detailed protocols to investigate thoroughly the effect of an altered medium viscosity on the growth rate and metabolism of both anchorage-dependent and freely suspended (hybridoma) cells. The early results are very promising, and show interesting effects of agitation and liquid shear on cell metabolism. This is part of an intensifying effort to better understand how the fluid-mechanical effects of agitation affect cell growth and physiology, and also investigates the different mechanisms that may be important in microcarrier-attached vs. freely suspended cells.

Studies continued on quantitating the effect of membrane shear stress imposed by well defined fluid flow on intracellular metabolism. The initial studies on fluid stress/metabolism coupling have utilized arachadonic acid metabolities as markers and have shown dramatic effects of flow on cell activation. Both anchorage dependent cells and cells in suspension have been employed. In addition, the PI has demonstrated a flow related increase in endothelial cell protein synthesis and secretion. Tissue plasminogen activator (TPA) was used as a marker protein. Over a period of 24 hours, flow generating a wall stress of 30 dynes/cm^2 lead to the secretion of about three times as much TPA as the no flow control.

An estimation and control study on a bioreactor with a remote sensor block was completed. A comparative study between linear and non-linear controller design showed that the non-linear controller provided a better performance. In addition, a parameter estimation scheme in which the dynamics of the parameters were modelled as random noises was implemented. With this scheme, process parameters can be estimated continuously on-line to improve controller performance. The potential of using an expert system to improve process performance is demonstrated through an experimental study of an expert system based pH controller. The results showed that the expert system based control scheme out-performed other existing scheme, including conventional linear PID controllers and modern adaptive controllers.

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Nucleation and Growth Control in Protein Crystallization

University of Alabama in Huntsville
Center for Microgravity and Materials Research

Dr. Franz Rosenberger
Dr. Edward Meehan
Dr. Thomas Nyce

Objectives: The inability to routinely grow crystals of sufficient size and quality is considered the major bottleneck in the further widespread development of the field of protein crystallography. By comparison with inorganic crystal growth, the fundamental understanding of the dynamics and kinetics of protein crystallization is still rather rudimentary. Correspondingly, the existing protein crystal growth technology provides only for limited control of essential growth parameters such as supersaturation and, consequently, the number and timing of nucleation events, and the growth rate. Most protein growth experiments to-date result in a large number of small crystals. In low gravity growth experiments these uncontrolled growth conditions can become somewhat mitigated. The reduction of buoyancy-driven, solutal convection reduces the probability for concurrent nucleation in the vicinity of initially formed crystallites. This may be one of the reasons for the increase in size and decrease in the number of protein crystals that have been obtained in space.

The objectives of this research program are: (a) contributions to the understanding of the dynamics and kinetics of protein crystallization in general; (b) the determination of the advantages and drawbacks of using temperature as the control parameter in protein growth; and (c) the development of a semi-automated protein crystal growth technology that excludes further nucleation after select initial nucleation or seeding, and provides for the controlled, non-interfering growth of the initial nuclei or seeds.

Research Task Description: Towards these objectives we are conducting (a) nucleation and growth experiments with lysozyme and porcine alpha-amylase solutions at low salt concentrations in temperature-programmable, optically accessable growth cells, in which information on nucleation and growth events is obtained with a simple light scattering and photodetection arrangement; (b) phase diagram work with lysozyme and alpha-amylase; (c) studies of protein growth and dissolution kinetics and morphology as a function of supersaturation through temperature variation, using interferometric microscopy with image storage and processing; and (d) model calculations on the diffusive transport dynamics of the hanging drop crystallization technique.

Progress to Date: The design and construction of a growth cell with light scattering setup, and with input into a PC-based temperature control/programming and data acquisition system has been completed. Tests of this system with the nucleation and dissolution (through temperature ramping) of alum solutions have been very encouraging.

The phase diagram work (solubility vs. temperature, salinity and pH) with lysozyme has revealed that the occurence of the orthorombic and tetragonal form is not solely determined by temperature and pH, but rather a combination of these parameters and ionic strength. In a few cases, however, it was found that the same combinations lead to either isomorph. Hence, there must still be some other, unidentified control parameter involved.

In preparation for the design of the in-situ optical growth kinetics studies we have found that surface morhological features, such as growth and etching steps, on protein crystals inside layers of solution can be observed with great image quality employing brightfield and interferometric microscopy.

The transport model calculations for the hanging drop technique have shown that, depending on the diffusivity (i.e. size) of the protein involved, significant protein concentration gradients can develop in the drop under purely diffusive conditions, whereas the drop will remain well mixed with respect to the salt concentration.

Publications

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Enhanced Hybridoma Production Using Electrofusion under Microgravity

University of Arizona, Tucson
Dr. David W. Sammons
NAG8-716
(NASA Contact: R.S. Snyder, MSFC)
May 1, 1988 - April 30, 1993

Objectives: Hybridoma technology and the production of monoclonal antibodies (MABs) has revolutionized biomedical research and medical diagnostics. MABs present new opportunities for the treatment of cancer, for the production of vaccines, and are essential for defining second-generation tests for the detection of life-threatening viruses such as Human Immunodeficiency Virus Types 1 and 2. It has been projected that the total MAB market will reach $6 billion by 1995.

Most MABs are derived from antibody-forming B-cells (usually the spleen of rodents) immunized with relatively large amounts of antigen and are immortalized by chemical fusion to suitable myeloma cells using polyethylene glycol (PEG). Antigen-specific hybridomas are selected from the many irrelevant hybrids that result from this fusion process by a variety of screening methods including immunoassay, western blotting and tissue staining. It is then necessary to sub-clone hybridomas previously selected to ensure their clonality and stability by any variety of labor-intensive methods and to repeat the screening prior to the "scale-up" for the production of the desired MAB. Using conventional methods, a period of six months may be required for a suitable hybridoma to be generated, selected, screened for specificity and stability, and for usable quantities to be made.

To date, overall efficiency and cost of production has proven adequate for most applications. Nevertheless, new applications (e.g., production of immunovaccines which are effective against rapidly mutating and poorly immunogenic viruses and immunotherapy of constantly changing malignant cells) will require large increases in the efficiency of antibody production and a substantial shortening of the time required for generation, selection and manufacture of suitable MABs. Moreover, the problems inherent in the use of xenographic MABs for "in vivo" applications (including short half-life, sub-optimal biological activity and the potential for life-threatening anaphylactic reactions) dictate the increasing need for human MABs. The production of useful human MABs is likely to be difficult for the following reasons: (1) suitable human B-cells are available in small numbers, (2) "in vitro" immunizations are required for the preparation of fusible human B-cells, (3) antigens to mutating viruses and changing malignant cells are likely to be limited and expensive to obtain, (4) as for murine hybridoma, today's time scale for production of usable quantities of human MABs will be excessively long, particularly for the purposes of immunovaccines production and anti-tumor therapy.

To meet the challenges of future application of MABs, considerable innovation is necessary to realize the time and cost reduction required in the hybridoma technology. Therefore, for these reasons, we are developing an extensive ground-based and microgravity program to modify methods and to implement emerging technologies in order to substantially improve the efficiency of and to expedite hybridoma production. Although our approach will exploit murine systems, we believe that this advanced technology will be readily transferable to the production of human hybridomas.

The long range objective of this cell fusion program is to attain high fusion

frequencies and hybridoma yields with methods that are rapid and efficient, and which are directly adaptable for future applications of the monoclonal antibodies. The primary mission of our effort is to perform ground based and microgravity research to improve electrofusion methodology and utilize the advantages offered by new technologies in B-cell selection and isolation which are required for the accomplishment of our long range goal. Our strategy is to pre-select antibody-producing B-cells prior to fusion (thus avoiding the need to select post-fusion selections), utilize in vitro immunization using anti-T-cell receptor complex MABs, utilize electrofusion instead of PEG, and conduct experiments under microgravity to advance our knowledge about the fundamental principles and processes involved.

Research Task Description: The prospect that the frequency of fusion and the yield of viable hybridomas is greatly enhanced under microgravity has been demonstrated with short duration flights of the TEXUS sounding rockets. We will be participating in the KC-135 and sounding rocket flights to extend previous findngs under microgravity and to test new protocols and hardware that are being developed during our ground based research. The work is directed toward optimizing electrofusion conditions and will utilize microgravity experiments (to be performed without sedimentation and convection forces) to advance our program goals. This research will culminate in a series of shuttle-based experiments designed to fully exploit the microgravity effects on cell fusion in the D-2 and subsequent missions.

To provide well-designed flight opportunities, the ground based research must address a number of defined and undefined cellular and physical limitations of the myeloma cell, the B-cell lymphocyte, the fused hyorid, growth and maintenance conditions, separation and evaluation methodologies and electric field conditions. Because of the multi-faceted nature of our cell fusion program, we are proceeding along several investigative lines in a simultaneous fashion. The current research tasks are as follows: (1) discover and develop optimal fusion partners for electrofusion, (2) develop optimal growth and maintenance conditions for parental and hybrid cells under "in vivo" conditions, (3) analyze cell populations and single cells for accurately monitoring cellular effects of the electric field conditions on the fusion process,
(4) separate and recover cell hybrids from the parent cells for determination of fusion frequencies and hybridoma yields, (5) recover cell organelles, membranes, and macromolecules of fused cells to determine the underlying changes that may be responsible for irreversible cell damage during execution of the fusion procedures, and (6) improve and innovate existing hardware for fusion of small numbers of cells under both ground based and microgravity conditions by taking into account the ground based and microgravity data.

This work is being performed in collaboration with Drs. Normal Klinman, Garry Neil and Howard Urnovitz, and in conjunction with Scripps Research Institute, University of Iowa, Calypte Biomedical Company (United States), and Drs. Kurt Hannig and Ulrich Zimmermann of the Max Planck Institute and the University of Wuerzburg (West Germany).

Publications

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Sharnez, R. and Sammons, D., "Resolving Real and Apparent Heterogeneity in Continuous Flow Electrophoresis," BioPharm Manufacturing, 1988 (submitted).

Schnettler, R., Gessner, P., Zimmerman, U., Neil, G. A., Urnovitz, H. B., and Sammons, D. W., "Increased Efficiency of Mammalian Somatic Cell Hybrid Production Under Microgravity Conditions During Ballistic Rocket Flight," J. Microg. Res., 1988 (in press).

Fluid Mechanics of Continuous Flow Electrophoresis

Princeton University
Dr. D. A. Saville
NAG 8-759
(NASA Contact: Dr. Robert Snyder, MSFC)
August 1986 - February 1989

Objectives: The work is a collaboration between Princeton and MSFC with the overall objective of establishing theoretically and confirming experimentally the ultimate capabilities of continuous flow electrophoresis chambers operating in an environment essentially free of particle sedimentation and buoyancy. The efforts at Princeton are devoted to: (i) improving the extant mathematical models to predict flow and particle trajectories in the apparatus as conceived at MSFC and (ii) a study of the effects of particle concentration on sample conductivity and dielectric constant. The dielectric constant and conductivity have been identified as playing crucial roles in the behavior of the sample and, thus, on the resolving power and throughput of continuous flow devices.

Research Task Description: Earlier work at Princeton (under contract NAS8-32614) showed that particle concentration has a strong influence on sample conductivity (more specifically, on the "real" or "dc" conductivity). Recent work has shown that the dielectric constant (the "imaginary" part of the complex conductivity) is similarly affected. Concurrently, experimental studies by P. H. Rhodes at MSFC showed that sample conductivity influences the spreading of the sample. A large conductivity mismatch between sample and the buffer causes the sample to spread rapidly from the front to rear walls of the channel. Rhodes developed an electrohydrodynamic theory of this spreading which shows that in addition to the conductivity, the dielectric constant should also affect spreading behavior. To optimize performance of a continuous flow device it will be necessary to understand the spreading process and, therefore, how it is influenced by the conductivity and dielectric constant. Accordingly we need to be able to measure the dielectric behavior of the sample as well as the DC conductivity.

The Princeton effort consists of three tasks:

1) Extension of existing mathematical models of flow and temperature fields in continuous flow chambers.
2) Development of a device to measure the conductivity and the dielectric contant of a suspension.
3) Theoretical work to relate buffer and sample compositions to conductivity and dielectric constant.

Progress to Date: Progress has been excellent and we can measure the properties of a given suspension at frequencies between 500 Hz and 200 KHz. A complete frequency scan takes about 25 minutes. The instrument has been used to measure the dielectric constant ad conductivity of latex suspensions similar to those used in Rhodes experiments. The results are linear in the volume fraction of particles for dilute systems (< 8 vol%) as they should be. One surprising results is that the low frequency limit is much larger (over 800 for a 4% by volume suspension) than the extant theory predicts In addition, the relaxation frequency is much lower than predicted by the theory.

Our results have been reported at several technical meetings and two papers accepted for publication in the Journa of Colloid and Interface Science. Inasmuch as this is the first time that the theory for the low frequency behavior has been subjected

to a rigorous test, our results have attracted considerable attention in the colloid science community.

It follows from these results that the effect of the dielectric constant on the sample's configuration may be even larger than anticipated. Clearly, it is of crucial importance to understand these phenomena if electrophoresis is to be used to fractionate cell or cell fragment populations.

Publications

Zukoski, C. F and Saville, D. A., "Electrokinetic Properties of Particles in Concentrated Suspensions: Heterogeneous Suspensions", J. Colloid Interface Sci, 1988 (accepted).

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Presentations

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Electrophoresis Technology

Marshall Space Flight Center
Dr. Robert S. Snyder
Percy H. Rhodes
In-House

Objectives: The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation; (2) characterize and optimize electrophoretic separators and their operational parameters; (3) develop innovative methods to accomplish electrophoretic separations in space; and (4) analyze the electrophoretic process using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separation.

Research Task Description: Both horizontal and vertical laboratory electrophoresis test chambers have been built to test the basic premise of continuous flow electrophoresis that removal of buoyancy-induced thermal convection caused by axial and lateral temperature gradients will result in ideal performance of these instruments in space. These gravity dependent phenomena disturb the rectilinear flow in the separation chamber when high voltage gradients and/or thick chambers are used, but distortion of the injected sample stream due to electrohydrodynamic effects causes major broadening of the separated bands observed in these chambers.

The electrophoresis separation process can be considered to be simple in concept but flows local to the sample filament produced by the applied electric field have not been considered. These electrohydrodynamical flows, formulated by G.I. Taylor in 1965 for drops suspended in various liquids, distort the sample stream and limit the separation. In addition, electroosmosis and viscous flow, which are inherent in the continuous flow electrophoresis device, combine to further disturb the process. Electoosmosis causes a flow in the chamber cross section which directly distorts the sample stream, while viscous flow causes a parabolic profile to develop in the flow plane. These flows distort the electrophoretic migration of sample by causing a varying residence time across the thickness of the chamber. Thus, sample constituents at the center plane will be in the electric field a different length of time and hence move more or less than comparable constituents closer to the chamber wall.

A moving wall concept is being developed for laboratory testing which will eliminate and/or control all of the above-mentioned disturbances. The moving wall will entrain the fluid to move as a rigid body and hence produce a constant residence time for all sample distributed across the chamber thickness. By aligning the moving wall at an angle to the chamber axis, a component of the moving wall motion can be made to oppose and hence cancel the electroosmotic flow. In absence of electrokinetic effects, i. e., electroosmosis, the electro-hydrodynamical effect manifests itself as a ribbon, being either vertical (perpendicular to the electric field) or horizontal (aligned with the electric field) depending on the ratio of conductivity of the sample to that of the buffer. Therefore, by using low conductivity sample solutions to provide a vertical ribbon, the moving wall concept should produce distortion-free separations.

Progress to Date: Future experiments will emphasize sample interactions of th type that can only be investigated in a free flowing system in the absence of buoyancy-induced thermal convection and sedimentation. The electrohydrodynamic distortion observed on STS-6 and STS-7 has now been modeled analytically and experiments that separately

consider electrical conductivity and dielectric constant discontinuities across the sample/buffer interface must be done. The model sample material will be selected to meet NASA program goals as well as the established electrophoresis operating parameters.

Publications

Rhodes, P. H., Snyder, R. S., and Roberts, G. V., "Electrohydrodynamic Distortion of Sample Streams in Continuous Flow Electrophoresis," J Colloid & Interface Sci., 1988 (in press).

Growth of DNA Crystals in a Microgravity Environment

University of Pennsylvania
Dr. Donald Voet
J. LaLonde
NAG8-611
(NASA Contact: K.D. Sowell, MSFC)

Objectives: The objective of the research is to determine the x-ray structure of several large segments of double stranded DNA. To this end it is necessary to crystallize the DNA. Since DNA is difficult to crystallize in a form suitable for X-ray diffraction, we are proposing to study the effects of microgravity on DNA crystallization.

Research Task Description: We intend to synthesize and crystallize several non-self-complementary segments of B-DNA in the rnage 12-20 base pairs. The crystals, when obtained, will be used to determine the X-ray structure of the DNA's in order to establish how the DNA conformation varies with its base sequence.

Progress to Date: We have synthesized seven different species of non-self-complementary double stranded DNA ranging in size from 12-20 base pairs. We have developed new methods of purifying these substances and have crystallized two of them: (1) a 12-base pair segment which is suitable for X-ray analysis, and (2) a 20-base pair segment which is yet too small for X-ray analysis but which we are hopeful of growing larger.

Capillary Convection with Crystal Growth

University of Michigan
Professor Wen-Jei Yang
NAG3-903
(NASA Contact: A.T. Chai)
May 1988 - May 1990

Objectives: Marangoni (surface tension) effects play a significant role in natural convection within a drop where crystal growth is induced by the "center-cooling" method. The physical phenomenon is, to a certain extent, similar to crystal growth from melts under microgravity. evaporating on a plate. It is proposed to study interfacial "turbulence" (namely Marangoni instability), internal flow structure and solidification front movement in the crystal growth of protein (such as lysozyme and myelin) and organic substances.

Research Task Description: The study is to obtain the on-ground information for exploiting the near weightlessness of space to grow protein crystals for the development of new drugs. The solidification process and its induced natural convection will be video-recorded with the aid of a microscope. The shadowgraph and holographic interferometry will be employed in the experimental study, while a numerical technique will be used in the theoretical investigation. The effects of surface tension-controlled natural convection on the growth of crystals will be determined. The crystal quality will be evaluated by Raman spectroscopy and compared with the quality of crystals grown under buoyancy-controlled natural convection. The study offers the possibility of understanding the origin, formation and eventual suppression of defects in crystal growth from melt or in fabricating new alloys under surface tension-controlled natural convection.

5. GLASSES AND CERAMICS

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Multimode Acoustic Research

Jet Propulsion Laboratory
Dr. Martin Barmatz
NAS7-918
October 1, 1981-September 30, 1989

Objectives: There is a recognized need for high temperature containerless processing facilities that can efficiently position and manipulate molten samples in the reduced gravity environment of space. The primary objectives of this task are to develop theoretical models of new classes of levitation, provide experimental validation of these models using research levitation devices, and investigate novel methods of sample levitation, manipulation, and heating.

The ultimate goal of this research is to develop sophisticated high temperature manipulation capabilites such as selection of arbitrary axes of rotation and rapid sample heating and cooling.

Research Task Description: The program tasks include the development of theories in uniform and temperature gradient environments for the acoustic forces and torques associated with sample translational and rotational stability using a variety of acoustic positioning modes (multimodes). These calculations are used to (1) determine those acoustic modes that produce stable levitation, (2) determine operating conditions to avoid translational and rotational instabilities, (3) determine the shape and position of levitated liquid drops, and (4) develop methods to translate levitated samples. This research task is also investigating the use of microwaves as a sample heating and positioning technique. Theories are being developed for the microwave heating and positioning of isolated samples. The application of microwaves to heat acoustically levitated samples will be demonstrated experimentally.

Progress to Date: Theoretical analyses carried out under this task have predicted stable acoustic levitation in rectangular, cylindrical and spherical resonators using one acoustic mode of excitation. This theory is now being used to design single mode levitators for use at very high temperatures ($\geq$1500 C). Stable levitation in a single mode cylindrical levitator has already been demonstrated in a ground-based laboratory up to 950°C.

Publications

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Patents

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Barmatz, M. and Garrett, S., "Stabilization and Oscillation of an Acoustically Levitated Object," Patent #4,723,266, September 1988.

Study of Powder Agglomeration in a Microgravity Experiment

Ohio State University
Professor James D. Cawley
NAG3-755 (NASA Contact: Dennis Fox, LeRC)
November 1986 - November 1988

Objectives: The population of agglomerates in the powder suspension plays an important role in the behavior of the system during shape forming processes. The ongoing research is designed to extend our understanding of the mechanistics of the agglomeration process through both experimentation and numerical modelling. In particular, we intend to exploit a microgravity environment to isolate convection from diffusion in the growth of agglomerates within dilute suspensions.

Research Task Description: In the first year of the program we concentrated on the use of optical microscopy to monitor the agglomeration of 0.4 and 4.0 m alumina particles constrained to the surface of an aqueous suspension. Through analysis of video tapes we have been able to identify that the relative contributions of convection and diffusion in the particle motion. The experiments suggest that the trajectory of the small agglomerates in a convective field is such that they preferentially deposit on the surface of larger agglomerates. This contributes to an unstable interface and ramified growth. The resultant agglomerates are fractal. The fractal dimension determined from an experiment on 0.4 m alumina was 1.55 in close agreement to that predicted using the cluster-cluster aggregation computer model.

Progress to Date: The experiments also showed that restructuring within the agglomerates was primarily due to the collapse of dendritic arms under the mild shear produced by convective flows. Efforts in this year have addressed the collapse of dendrite arms in both experiments and modeling. One of the difficulties in interpreting the restructuring is that the yield stress is determined by the surface irregularities on the nearly spherical alumina particles. In order to obtain a situation in which there yield stress is more predictable we have synthesized cubic hematite particles following the procedure of Matijevic and coworkers. Experiments to date indicate that the structure of the agglomerates is only weakly affected by the shape of the primary particle, that is, the results from the hematite are very similar to those from alumina. Rearrangement due to convection is also being addressed in the modeling using Brownian Dynamics. The activation energy for rearrangement arises because rotation causes the surface of the particle to separate. Inclusion of this factor in Brownian Dynamics therefore requires the interaction energy to be known for arbitrary particle orientations. Unfortunately expression for van der Waals interactions exist only for simple geometries. We have been partially successful in obtaining an empirical solution based on pairwise interaction of spheres filling a cubic volume. Also, some effort was expended to develop a routine based on a discrete fluid treatment. This was motivated by recent report of convective flows being observed in hard disk fluids. We are no longer actively pursuing this approach.

The experiments have been refined by improving the uniformity of the illumination which greatly aids in the accuracy of digitized images.

Glass Formation in Reluctant Glass Formers

Marshall Space Flight Center
Dr. Edwin C. Ethridge
In-House

Objectives: The objective of this research is to investigate the crystallization kinetics and glass forming ability of reluctant glass formers. This could ultimately aid the formation of bulk samples of unique glass compositions outside of normal glass forming regions allowing the optimization of certain properties of the glass.

Research Task Description: An important aspect of processing in space is the containerless undercooling of molten substances. Theoretically, the extent of undercooling can be greatly enhanced by solidifying in the absence of heterogeneous nucleation resulting from contact with crucibles or molds. The containerless solidification of reluctant glass formers may permit much slower cooling rates to form glasses than is otherwise required.

Progress to Date: This work has concentrated on establishing techniques for the measurement of crystallization kinetics and critical cooling rates. In the absence of suitable 1-g levitation melting techniques, a thermocouple was used for sample support. Recently the effects of various processing conditions on the heterogeneous nucleation on an eutectic composition within the gallia-calcia system were reported. The experiments utilized an ellipsoidal heater to melt the samples which were contained on a type *S* thermocouple positioned at one foci of the ellipsoid. The sample thermal history was recorded directly by a strip chart recorder and the experiment was visually recorded and imaged and recorded with a video camera/recorder. Under most experimental conditions the cooling rate is approximately linear. Many hundreds of experiments were performed. As a result f the research it was found that processing conditions affect sample nucleation in various ways. Some of the more important processing conditions are the extent of superheating above the thermodynamic melting temperature and the mechanism of quenching the sample.

The apparatus for nucleation experiments is being automated. Obtaining data utilizing current techniques is very time consuming and labor intensive, but automation of the experiments will greatly increase experiment efficiency. Experiment automation combined with telepresence and telescience are future directions for some of the materials processing space experiments. Schmidt et (1987) presented arguments for the promotion of telescience as the operational philosophy of future space experimentation. Based upon prior experience with Spacelab, there are typically about three (3) hours of astronaut crew time available for Space Shuttle based manned operations of each experiment rack per day. On Space Station, however it is projected that there will be much less time for crew tending of experiments being on the order of 20 minutes per experiment rack per day. This low level of astronaut availability may require that many experiments be highly automated. Also, it is likely that flight opportunities may exist for experiments on free flying man-tended satellites such as the Industrial Space Facility (ISF). Ideally each experiment could be operated from ground based laboratories in which the principal investigator could select processing conditions, initiate the experiment, and view the experiment (both data and video) in a near real time mode. Nucleation and crystallization experiments are well suited to automation and the development of these telepresence and telescience concepts on earth. Aspects of telescience are being incorporated into the automated apparatus.

Publications

Ethridge, E. C. and Curreri, P. A., Heterogeneous-Nucleation and Glass-Formation Studies of $56Ga_2O_3$-44CaO," J. Am. Ceram. Soc. 70, 553-556 (1987).

Study of Foaming in Glass Melts Under Microgravity

Case Western Reserve University
Dr Pavel Hrma
Dr. Ali Ilhan
NAG3-740 (NASA Contract: M. Jaskowiak, LeRC)
October 1, 1987 - October 1, 1988

Objectives: The main objectives of the research is to remove external body forces during free fall, obtain a better understanding of the role of gravity in the behavior of transient foams in glass melts and room temperature model liquids. This involves development of a model for generation and collapse of transient foam, selection of suitable materials, and establishment of an appropriate experimental technique to determine the difference in foam behavior under earth gravity and microgravity.

Research Task Description: The first step is to identify grvity sensitive melts and room temperature model liquids that produce transient foams with collapse rates compatible with the time available during the free fall in a drop tower. The second step is to develop a technique for reproducible foam generation and collapse such that a clear comparison between foam behavior under earth's gravity and reduced gravity would be possible. The following requirements should be met: (1) sufficient degree of collapse during the 5 sec. of the free fall in the drop tower experiment; (2) narrow bubble size distribution; (3) excellent reproducibility of foam behavior; (4) simple design, manipulation, and adaptability of the experimental set-up. Both melts and room temperature model liquids were investigated to identify suitable systems for drop tower experiments. Transparent (quartz glass) containers were used for melts and teflon coated glass containers for room temperature liquids. A transient aqueous foam was generated from water containing surfactant and oversaturated with carbon dioxide. Foaming was triggered either by reducing pressure or addition of solid particles and recorded by a video camera.

Progress to Date: A previously initiated model for the steady state foam blanket was completed. Simple models for collapse of foam generated by injection or entrapment of external gas and for generation and collapse of foam arising due to a gas liberating reaction within the liquid phase were developed. In these models, the collapse coefficient, which can be evaluated from experimental data, is expressed in terms of the size of the foam cell, its residence time at the foam surface, and the foam height. In addition, conditions for development of bulk foam and surface foam were specified and criteria for applicability of drop tower to foam dynamics studied were established.

Foams in glass melts and molten inorganic salts other than those produced by decomposition of sodium sulfate were found gravity insensitive or extremely unstable. Foam in soda-lime-silica glas with sodium sulfate as a foaming agent was produced under atmospheric pressure by increasing temperature and under constant temperature by reducing pressure. The collapse time under atmospheric pressure at $T \geq 1450$ C was ~3 min; the residence time of bubbles at the foam surface was $\leq$ 1s, which enables observation of changes in collapse rate during 5s of the free fall. Three methods of foam generation in room temperature liquids oversaturated by carbon dioxide were tested: gas entrapment, pressure decrease, and addition of solid particles. So far the best reproducibility of foam height vs time curves in room temperature model liquids was obtained by addition of particles of well-defined shapes and surface area under atmospheric pressure. Particles that were rough or porous (sintered alumina, porous ceramics), polydisperse (silica sand), water soluble (sugar, rock salt), or very fine (less

than 500 μ m) produced less reproducible foams. Also, uncoated or freshly coated containers exhibited poor reproducibility. Furthermore, the way of pouring the liquid into the container and introducing solid into the liquid affects reproducibility. Gas entrapment produced foams with nonuniform bubble sizes. Pressure decrease caused frequent coalescence. Experimental set-up for drop tower study will contain a battery of five transparent rectangular containers, in which foam will be started in adjustable time intervals to cover the time range of foam collapse during the free fall.

Publications

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Study of Phase Separation in Glass Under Microgravity

NASA Lewis Research Center
Mark J. Hyatt
In-Center

Objectives: The objective ofthis research is to define an experiment which will use a reduced gravity environment to aide in the study of phase separation in glass systems. In a reduced gravity environment, surface forces will dominate the separation of phases. This will enable study of the fundamental aspects of phase separation.

Research Task Description: The research task can be decribed by four steps:

1) Measure physical property data of a model glass system.
2) Modeling of phase separation process.
3) Predict behavior of glass system using phase separation model.
4) Define low gravity experiment which will determine actual behavior of the glass system and compare to model predictions.

Progress to Date: A phase equilibria study of the ternary bismuth borosilicate system has been conducted. The complexities of studying a ternary system will probably lead to selection of the binary bismuth borate system as the model glass system. Crucible corrosion, melt volatization, and layering kinetics were also studied for both ternary and binary compositions.

Glass Research

Jet Propulsion Laboratory
Dr. George F. Neilson
Dr. Michael C. Weinberg, University of Arizona
In-Center

Objectives: The overall objective of this program is to establish the scientific framework for the identification and evaluation of potential flight experiments pertaining to the preparation and processing of glasses in a microgravity environment by means of ground-based experimentation and mathematical modelling. In pursuit of this objective, the following are the current specific tasks: (1) study the feasibility of conducting a glass immiscibility flight experiment; (2) conduct a computer analysis of complex T-T-T diagram behavior including calculation of volume fraction crystallized to allow comparison of ground-based and microgravity glass crystallization behavior; and (3) compare the homogeneity and crystallization behavior of gel-derived glasses prepared by different processes to those of conventional glasses of identical compositions.

Under task 1 the unique advantages of conducting a containerless flight aamorphous phase separation experiment in the lead borate system is under study. Suitable compositions are being sought where it can be determined whether -g will offer a unique advantage to study the process due to elimination of gravity-driven density phase segregation. Under task 2 T-T-T curves are to be generated by computer simulation from evaluation of the JMA equation for model systems. Account is to be taken of the possibilities of combined homogeneous and heterogeneous nucleation, saturation, and transient effects. The volume fraction of crystals formed under both isothermal and nonisothermal conditions will be investigated experimentally and theoretically. Under task 3 the microstructural behavior of gel-drived and conventional glasses in the sodium borosilicate system are being studied by combination of optical microscopy, SAXS, and IR spectroscopy. The effect of gel structure on ability to form homogeneous glass will also be studied.

Progress to Date: It was determined that a composition regime in the lead borate system exists which has a suitable viscosity range to allow unique μ-g immiscibility kinetic studies to be conducted. These results will be incorporated as part of a flight proposal to determine the role of gravity in the phase separation kinetics of fluid glass melts. Equations were derived and employed to allow comparison of the relative importance of saturation effects on both homogeneous and heterogeneous nucleation. Several possible saturation mechanisms which effect nucleation processes were investigated.

A new analysis was conducted to compare the experimentally determined with the theoretically predicted temperature dependence of the homogeneous crystal nucleation rate. It was concluded that the temperature dependence given by classical theory is still a matter of controversy.

Several sodium borosilicate composition glasses near the edge of the immiscibility region were prepared by conventional and gel methods to compare glass homogeneity as a function of melting time. Preliminary optical and SAXS data were obtained. Plans were made to transfer the Glass Research Lab facilities and personnel from JPL to the Materials Science and Engineering Department of th University of Arizona in September.

Publications

Weinberg, M. C., and Neilson, G. F., "A Comparison of the Phase Transformation Behavior of Gel-Derived and Ordinary Soda-Silica Glasses," in *Sol-Gel Technology* (L. Klein, ed.), 1988.

Zanotto, E. D., and Weinberg, M. C., "Saturation Effects in Homogeneous and heterogeneous Nucleation," J. Non-Cryst. Solids, 1988 (in press).

Weinberg, M. C. and Zanotto, E. D., "Re-examination of the Temperature Dependence of the Classical Nucleation Rate; Homogeneous Crystal Nucleation in Glass," J. Non-cryst. Solids, 1988 (submitted).

Spherical Shell Technology

Jet Propulsion Laboratory
Dr. Taylor G. Wang
J. M. Kendall
M. Chang
C. P. Lee
M. C. Pee
M. Zak
In House

Objectives: The objectives of this task are to study the rotational and vibrational behavior of free liquid shells of varying aspect ratios. Mode splitting and coupling due to the gaseous coreand the core centering mechanics will be of special interest.

Research Task Description: The science and technology of a new method for the production of spherical shells are developed under a coordinated experimental and theoretical program. The underlying production process is fluid-dynamic in nature and requires low or zero gravity. Foreseeable applications pertain to lightweight structural materials, to biotechnology, and to a variety of technical or industrial products.

The gravitational effects and the hydrodynamic instability with inclusion of the effects of viscosity, rotation, etc., are being studied. One emphasis of the work is encapsulation of biological materials for human implantation. Here cells or other biological materials are encapsulated within 200-400 μ m shells of specified permeability. Nutrients are free to pass into the shells, reactive products are fee to diffuse outward, but the cell within each is protected against the large molecular-weight compounds of the immune system borne by the host. Accomplishments include the production of shells of uniform size of various polymers, a demonstration permeability to small and medium-sized molecules, and a demonstration of impermeability to large molecules. Also, encapsulation of cell-sized inert particles has been accomplished.

Progress to Date: Theoretical model for the annular jet instability has been developed in which the liquid layer enclosing the gaseous stream in the jet is modeled as a membrane with no thickness but finite mass/area which moves under the influences of its own surface tension, inertia, and the gaseous pressure. If should be noted that the hollow jet instability is phenomenon completely different from the more familiar Rayleigh instability of a simple jet. The methodology of converting a free surface flow problem into a one-dimensional one using a thin sheet model that can be handled more easily is an innovative contribution to the field of theoretical nonlinear physics.

We have derived a microscopic model of first-order phase changes in a one-component system with spherically symmetric, two-body potentials. Included is a microscopic model for the driving forces inducing the phase change, cooperative model for homogeneous nucleation, and a possible solution to the "fcc-hcp" problem in rare gases.

The basis of the production method involves the extrusion of liquid through suitable nozzles, following which discrete, precision, droplets are formed. These solidify in spherical and concentric form by means of freezing or by chemical reaction without gravitational distortion. The research concerns the hydrodynamic instability of liquid or compound jets, with inclusion of the effects of viscosity, rotation, etc. Both numerical and analytical results are utilized to interpret the experimental findings. Current

emphasis of the work is upon encapsulation for biotechnology applicants. Here, cell or other biological materials are encapsulated within 200-400 μ m shells of specified permeability. Nutrients are free to pass into the shells, reactive products are free to diffuse outward, but the cell within each is protected against the large molecular weight compounds of the immune system borne by the host. Accomplishments include the production of shells of uniform size of various polymers, a demonstration of permeability to small- and medium-size molecules, and a demonstration of impermeability to large molecules. Also, encapsulation of cell-sized inert particles has been accomplished.

Publications

Chang, M., Kendall, J. M., and Wang, T. G., "A Novel Technology for the Micro-encapsulation of Living Cells," J. Biotechnology, 1988 (submitted).

Wang, T. G., "The Theoretical Model for the Annular Jet Instability - Revisited," Phys. Fluids, 1988 (submitted).

Glass Forming and Crystallization in Space

University of Arizona
Dr. M. C. Weinberg
Dr. G. Neilson
(NASA Contact: G. Neilson, JPL)
September 1987 - September 1991

Objectives: The objectives of this research are: (1) to study bulk (homogeneous) crystal nucleation in lithium diborate and other simple glasses; (2) to assess the viability (and desirability) of performing such nucleation experiments in a containerless facility in space; (3) to provide accurate methods of computing the volume fraction crystallized in complex systems and/or systems of small particles; (4) to investigate the surface characteristics and surface devitrification tendencies of lead borate glasses; (5) to study crystal growth rates in high PbO composition lead borate glasses; and (6) to assess novel optical and electro/optical applications of $PbO-B_2O_3$ glasses containing large proportions of lead.

Research Task Description: Crystal nucleation may be enhanced by the presence of foreign substances in contact with the melt, leading to heterogeneous nucleation. Since crucible walls and contaminants introduced into the melt from the crucible can serve as heterogeneous nucleation sites, and uncontained melt might be subject solely to homogeneous nucleation. It is this belief which is the basis for the anticipated benefit of containerless processing for the potential production of novel glass compositions.

However, in order to be able to assess the potential advantages of containerless processing two items are required: (1) comparative ground based nucleation and crystallization experiments, (2) a more comprehensive knowledge of the factors which influence crystallization processes and a reliable theory to explain the latter. It is the purpose of this program to provide a framework which will allow for the interpretation, and guide in the judicious selection, of glass flight experiments pertaining to glass crystallization.

A unique opportunity for studying glass formation and for the possible extension of the normal glass-forming compositional range is offered by a containerless processing facility in a microgravity laboratory. It is important to select appropriate compositions for such studies. It is believed that $PbO-B_2O_3$ compositions are well suited for this purpose for the following reasons. Lead glasses have a wide variety of commercial applications, and are of scientific interest, too, since they exemplify one of the unusual cases where large concentrations of a non-traditional glass-forming cation (pb) can be present in a glass-forming composition. Other advantages include: low liquidus temperature, acceptable degree of volatility, and a demonstrated tendency to devitrify at the melt-container interface.

In order to provide a framework for the interpretation of flight experiments, two types of ground-based experiments are planned at the University of Arizona. First, crystal growth studies will be executed as a function of temperature and glass composition. Next, the relative proclivity for free surface vs. container induced surface devitrification will be assessed. These experiments are key for understanding the intrinsic glass-forming limits of these compositions.

Progress to Date: Two approaches were employed to calculate the non-isothermal crystallization of undercooled liquids on both heating and cooling paths. The first used

the concept of additivity and the second used a rigorous expression. It was indicated that the additivity approximation is strictly valid in two limiting cases: (i) site saturation or (ii) complete overalp of nucleation and growth curves. Numerical calculations were performed for SiO_2 glass, which has well-separated nucleation and growth curves, and significant differences (orders of magnitude) were found between the use of the two methods for the prediction of the volume fraction crystallized. Despite this discrepancy, the calculated critical cooling rates differ by less than an order of magnitude. It was also demonstrated that the overall crystallization produced on heating is many order of magnitude higher than that on cooling (for equal dT/dt) due to the fact that the maximum nucleation rate occurs at a much lower temperature than the maximum crystal growth rate.

A comparison was made between the experimentally determined and predicted (Classical Nucleation Theory) temperature dependence of the homogeneous crystal nucleation rate in glass. It was shown that the standard assumptions which are invoked to replace the free energy of activation for transport across the liquid-crystal interface, ΔG_D, in favor of a more easily measured parameter (viscosity) are not required. It was demonstrated that ΔG_D may be found experimentally by determining the temperature dependence of the nucleation transient time. It was concluded that the temperature dependence of the classical homogeneous nucleation rate is still a matter of controversy.

Publications

Uhlmann, D. R., Weinberg, M. C., and Teowee, G., "Crystallization of Gel-Derived Glass," J. Non-cryst. Solids 100, 154 (1988).

Zanotto, E. D. and Weinberg, M. C., "Saturation Effects in Homogeneous and Heterogeneous Crystal Nucleation," J. Non-cryst. Solids, 1988 (in press).

Weinberg, M. C. and Zanotto, E. D., "On the Calculation of the Volume Fraction Crystallized for Non-Isothermal Transformations," Phys. Chem. Glasses, 1988 (in press).

Weinberg, M. C. and Zanotto, E. D., "Re-examination of the Temperature Dependence of the Classical Nucleation Rate: Homogeneous Crystal Nucleation in Glass," J. Non-cryst. Solids, 1988 (accepted).

6. COMBUSTION SCIENCES

The Combustion of Free or Unsupported Fuel Droplets at Low Gravity

Cornell University
Professor C. Thomas Avedisian

Objectives: This research will study the combustion of free or unsupported fuel droplets in a low gravity environment. Single component and multicomponent fuels will be studied. The evolution of droplet diameter, total burning time, flame shape, microexplosion (if it occurs) characteristics, soot particle structure (for sooting fuels), and extinction diameter (if extinction occurs) will be measured.

Research Task Description: The experiments will be carried out in a small scale (7.6m) drop tower. The initial droplet diameter will be about 500 m, which is small enough that the complete burning history can be recorded for the experimental time available (about 1.3 seconds). An experiments will be initiated by simultaneously releasing the test droplet, its enclosed environment (i.e., combustion chamber containing the oxidizing gas), and instrumentation package into free fall. The principle means of data acquisition will be photographic. During the period of fall, the droplet burning history will be recorded by one or more on-board cameras: both high speed cine and video cameras will be used.

Fuel Droplet Vaporization in a Supercritical Environment at Microgravity Conditions

University of Wisconsin-Madison
Professor Gary Borman
Professor P. V. Farrell
Professor P. S. Myers
Professor D. E. Foster
Dr. D. L. Reuss, GMRL
NAG3-718
(NASA Contact: Kurt Sacksteder, LeRC)
April 15, 1986 - April 14, 1988

Objectives: The major objective of the research is to study single droplet vaporization under conditions at or above the critical temperature and pressure of the pure liquid. The problem is of considerable interest in high pressure liquid fuel combustors, such as liquid fuel rockets, and compression-ignition (diesel) engines.

The research will attempt to answer questions regarding droplet vaporization rate, surface tension effects, transport property anomalies, and dynamic effects due to near-critical conditions. In order to minimize the significant influence of convection (free and forced) in a non-isothermal field, a microgravity environment is required.

We are currently developing a model and experiment for addressing the objectives of the research.

Research Task Description: The research tasks maye be divided into modeling related tasks, and experiment related tasks.

The experiment-related tasks consist of a number of activities which will help produce a device which can place a motionless liquid droplet in a known location in a device which will then rapidly raise the atmospheric temperature and pressure to values greater than te critical temperature and pressure for the liquid. Diagnostics will recird data regarding the subsequent vaporization of the droplet. Sub-tasks include developing the drop-deployment device, developing the compression device, developing appropriate synchronization controls, and developing diagnostics. Since th device must be tested in microgravity, all portions must work in a stand-alone micro-g environment, and withstand a high quality environment at the end of the experiment.

The modeling Task involves modeling the droplet vaporization problem as a 1-D diffusion problem. The primary difficulty in the modeling is developing an equation of state appropriate for use over a wide range of temperature and pressure, including supercritical values. The modeling will simulate the behavior of the entire experiment from drop formation through compression and vaporization.

Progress to Date: The compressions device is essentially complete and has been dropped about 20 times at the NASA Lewis Research Center 2.2 second drop tower, using a high speed movie camera to record droplet behavior. The device, including timing circuits and instrumentation seems to work well and is quite rugged. The droplet deployment device mounted on the compression device has not been quite as successful and work is continuing on making repeatable low velocity droplets. An on board computer has been built to control the sequencing of the various devices, as well as provide 8 channels of about 4kHz 12 bit A/D conversion. A Rayleigh scattering system for measuring gas phase species concenteation is being developed using a ruggedized laser and fiber optic

probe. Considerable effort has aso been expended in investigating systems capable of non-intrusively positioning the droplet, if low-velocity deployment cannot be achieved. We have successfully positioned solid spheres (~ 30 μ m) and liquid droplets (20-40 μ m) using optical levitation techniques.

The modeling effort has produced working models for several liquid-gas pairs (octane-nitrogen, decane-nitrogen). In addition, several potential equation of state calculations have been evaluated to deterine the most accurate for our application. Work continues on improvements to the model as well as expanding the liquid-gas pairs.

Publications

Curtis, E. W. and Farrell, P. V., "Droplet Vaporization in Supercritical Microgravity Environment," Acta Astron., 1988 (in press).

Curtis, E. W., Hartfield, J. P., and Farrell, P. V., "Microgravity Vaporization of Liquid Droplets," in *Proceedings of Third Colloquium on Drops and Bubbles*, Monterey, CA, 1988 (submitted).

Buoyancy Effects Upon Vapor Flame and Explosion Processes

Science Applications International Corporation
Dr. Raymond B. Edelman*
Dr. M. Yousef Bahadori
NAS3-22882 (NASA Contact: Dennis Stocker, LeRC)
November 1984 - January 1989

Objective: The objective of this microgravity project is to gain a better fundamental understanding of the effects of buoyancy on laminar gas jet diffusion flames and to establish the relationship between buoyancy and (1) unsteady phenomena associated with ignition and flame development, (2) steady-state flame structure, (3) soot formation and disposition, (4) radiation, (5) extinction phenomena, and (6) chemical kinetics. The findings will aid in defining the hazards and control strategies of fires in spacecraft environments as well as to improve the understanding of earthbound fires. The specific objectives of the program are to: (a) obtain measurements that include flame-shape development, flame extinction, flame color and luminosity, temperature distributions, species concentration, radiation, pressure, and acceleration, and (b) extend the numerical models developed to date to include transient effects, chemical kinetics, soot formation, and more detailed radiation effects.

Research Task Description: The program is structured in terms of closely interrelated ground-based experiments and theoretical modeling. This program has evolved as a result of theoretical analyses and limited experimental observations which have delineated the requirements to gain a more fundamental understanding of the effects of buoyancy on gas jet diffusion flames. The experimental portion of the ground-based program is designed to provide both additional time and quantitative measurements based on the findings of the past 2.2 second drop-tower experiments. These data will be obtained by the combined use of the Lewis 5.18-sec. zero-gravity facility and the KC-135 aircraft. The results will be used as a database for the model development and will clarify the requirements on time for approach toward steady state. This will establish the need for space experiments.

Progress to Date: Progress to date includes various analyses and modeling for the support of experiment fabrication. Work in progress includes the development of steady-state and transient models, in addition to the selection and development of submodels for diffusion, gas-phase kinetics, radiation, and soot generation and burn-off for inclusion in the models. The results of the 2.2-sec. drop-tower experiments have shown that hydrocarbon flames can be successfully ignited in microgravity, using a spark ignitor. In addition, fabrication of the experiment package is near completion. The various findings of the experimental and theoretical efforts have been recently presented at the AIAA 26th Aerospace Sciences Meeting, Reno, Nevada, January 1988) and Second Symposium on Lunar Base and Space Activities of the 21st Century (Houston, TX, April 1988).

Publications

Edelman, R. B., Bahadori, M. Y., Olson, S. L., and Stocker, D. P., "Laminar Diffusion Flames under Microgravity Conditions," *AIAA 26th Aerospace Sciences Meeting*, Reno, Nevada, January 11-14, 1988, Paper AIAA-88-0645; AIAA J. (submitted).

Bahadori, M. Y. and Edelman, R. B., "Gas Jet Diffusion Flames under Reduced-G Conditions," in *Proceedings of Second Symposium on Lunar Bases and Space Activities of the 21st Century*, Houston, Texas, April 1988 (in press).

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A Fundamental Study of the Effect of Buoyancy on the Stability of Premixed Laminar Flames

University of California at Berkeley
Professor A. Carlos Fernandez-Pello
Corey M. Dunsky
NAG3-861
(NASA Contact: D. Stocker, LeRC)

Objectives: The objective of this research is to investigate the effect of buoyancy on the stability of premixed laminar flames. The information obtained will help in the understanding of the mechanisms responsible for instabilities in laminar flames which are often regarded as precursors of turbulent combustion processes. The immediate objective is to perform a set of low-gravity, ambient and low pressure drop-tower experiments to elucidate the effect of the absence of gravity on the onset and evolution of cellular flame structure.

Research Task Description: The research task consists of the experimental determination of the gravity effect on the stability and cellular structure of a burner-stabilized premixed flame. Normal gravity-atmospheric pressure experiments are first performed to identify the range of parameters at which the flame becomes unstable and the cellular flame structure appears. These experiments are followed by low gravity-atmospheric pressure tests performed at the 2.2 sec NASA LeRC drop tower. The results for the normal and microgravity tests are compared to observe, through the cellular structure, the effect of gravity on the flame stability. These experiments are complemented with a similar series of tests performed at varied ambient pressure. Since the ambient pressure affects the buoyancy force, diffusivity and reaction rate, the comparison of the different results are used to determine the effect of gravity on the individual mechanisms of flame stabilization.

Progress to Date: During this reporting period work has concentrated on the design, construction and laboratory testing of a new drop tower package. The package will be used at the NASA Le RC facility to conduct additional tests on the cellular flame structure under microgravity conditions. An important feature of the new apparatus is the accurate control of the fuel/oxidizer mixture in the burner. This is accomplished by using electronically controlled mass flow meters which are used to either meter the flows going to the burner or to prepare a predetermined gas mixture for it later use in the burner. The package is currently near completion and tests are currently underway to map with the new meters the fuel/oxidizer flow ranges at which cellular flames are observed. Drop-tower tests are scheduled for the months of November and December, 1988.

Concurrent with this work, an experimental study is being conducted to investigate the effect of ambient pressure on the stability and the structure of cellular premixed laminar flames. The objective of the study is to complement the investigation of the effect of gravity on cellular flames. The ambient pressure not only affects buoyant forces, through the density difference, but also affects other parameters that influence the flame structure. Therefore, the results of this study will aid in the identification of the dominant mechanisms responsible for the flame instabilities.

Publications

Dunsky, C.M. and Fernandez-Pello, A.C., "The Effect of Ambient Pressure on the Cellular Flame Structure," *1988 Fall Meeting. Western States Section/The Combustion Institute*, Dana Point, CA, October 1988.

Radiative Ignition in Microgravity Environment

National Institute of Standards and Technology
Dr. Takashi Kashiwagi
Dr. Howard R. Baum
Dr. Colomba di Blasi

Objectives: The objective of this study is to develop a theoretical model capable of predicting the radiative ignition of a cellulosic material (for example, filter-paper) and subsequent flame spread over the sample in microgravity environment. This study is to lead to applications such as fire safety for spacecraft by understanding and predicting ignition and subsequent flame spreading behavior. It is hoped that validation experiments of the predicted results will be conducted in microgravity environment in the next phase of the study.

Research Task Description: This study consists of four parts: (1) Thermal and thermal oxidative characterization of the cellulosic material will be measured in a normal gravity environment by using a thermal analysis technique. A global degradation scheme involving two or three step reactions will be used and their kinetic constants will be determined by dynamic derivative thermogravimetry at various heating rates and also at various ambient oxygen concentrations; (2) Kinetic constants of global gas phase oxidation reactions of evolved degradation products from the degrading cellulosic material will be determined in a normal gravity environment by using a well-defined laminar high temperature flow reactor; (3) A theoretical model of ignition of the cellulosic material by an external radiation and subsequent transition to flame spreading in a microgravity environment will be developed and solved numerically using the above characteristics of the degradation and of the global gas phase oxidation reactions. By using the advantage of a microgravity environment is fully utilized (the dominant vorticity creation mechanism in the bulk of the gas is absent), the gas phase flow pattern is calculated by solving an irrotational flow pattern determined by the thermal expansion of the field and also convective mass transfer of the evolved degradation products to the gas phase; (4) Radiative ignition experiments at a normal gravity environment will be conducted to examine ignition behavior and also to obtain radiative properties of the cellulosic material. The bottom surface of the sample will be ignited by a vertically upward external radiation. Ignition behavior in this configuration in a normal gravity environment is the closest to that in a microgravity environment.

Progress to Date: The flow pattern and the temperature distribution in the gas phase during the preheating period, which is controlled by a balance between the thermal expansion of the gas due to the heat addition from the heated surface to the gas phase and conduction away from the heated region, were calculated first. The results were obtained analytically for early time and numerically for most times of interest in the experiments envisaged for this study. The additional complexity of convective mass transfer of the evolved degradation products from the sample surface to the gas phase is currently being incorporated into the preheating model. The characterization of thermal and thermal oxidative degradation of the cellulosic material is on progress including the measurements of CO, CO_2, and H_2O in the evolved gases. The construction of an ignition chamber for radiative ignition experiments in a normal gravity has been completed. The calibration of the radiant flux distribution and preliminary ignition experiments are being conducted. Shadowgraph photography set up to observe ignition events is being assembled. The overall design of the flow reactor is completed and some of its components, for example high temperature flow settling chamber, are being constructed.

Presentations

Baum, H., Kashiwagi, T., di Blasi, C., "Radiative Ignition of Solid Fuels in a Microgravity Environment - The Preheating Problem" *1988 Eastern States Combustion Meeting*, December 1988.

Effect of Low Velocity Forced Flow on Flame Spread Over a Thermally-Thin Solid Fuel in the Absence of Buoyancy-Induced Flows

NASA Lewis Research Center
Sandra L. Olson
In-House

Objectives. The objective of this study is to determine the effect of low velocity opposed forced flow on flames spreading over thermally-thin fuels. The approach used in this study is to perform a series of experiments in low gravity varying oxidizer concentration and opposed flow velocity to determine the extinction limits and steady burning characteristics of flames spreading over solid fuels under these conditions. Tests will be conducted in a low speed combustion tunnel developed for use in all three NASA Lewis Research Center's low gravity facilities.

Low gravity is required for these experiments because in normal gravity buoyancy-induced gas flows around the spreading flame are on the order of or greater than the range of forced flow velocities to be studied (0-30 cm/s). These natural convective flows overwhelm or combine with the forced convective flows so that the effect of the forced flow on the flame spread rate cannot be isolated.

Research Task Description: The study will be performed in four phases: 1) Preliminary normal and low gravity quiescent environment (zero flow) and normal gravity high velocity forced flow experiments will be performed to define fuel burning characteristics and to estimate fuel extinction limits as a function of oxygen concentration and flow velocity; 2) simultaneously, flow field characterization of the combustion tunnel with cold flow in normal and/or microgravity will be performed, and instrumentation will developed for the low gravity tests; 3) normal gravity tests and low gravity tests in three Lewis low gravity research facilities will be conducted to determine the extinction limits as a function of oxygen concentration and flow velocity. The flame development and steady burning characteristics of flame spread over solid fuels will be studied and compared with current modelling work underway at Lewis; and 4) when the ground-based research is completed, the data will be analyzed and a report published of the results. If it appears that a space experiment is appropriate and feasible, a Science Requirements Document will be drafted. If necessary, further ground-based research will be proposed.

Progress to Date: The 2.2 second Combustion Tunnel Drop Tower package was completed in February 1988, and the supporting flow system was completed in March 1988. Flow visualization methods, pressure transducer calibrations, and normal gravity qualitative flow visualization experiments were conducted from April to July, 1988.

Instrumentation to measure solid and gas phase temperatures during a drop were developed in preparation for the combustion experiments which will begin after the flow calibration work is completed. The quiescent environment flame spread and extinction experimental results completed in 1987 were presented at The Twenty-Second Symposium (International) on Combustion in August 1988.

Quantitative flow visualization revealed a strong acceleratory effect of buoyancy from the hot wire used to generate smoke, especially at the lower flow rates, so in September 1988 a microgravity test program was initiated to conduct the quantiative flow visualization in microgravity and thus eliminate the buoyancy effect. The microgravity flow calibration is expected to continue through the remainder of 1988.

Publications

Olson, S. L., Ferkul, P. V., and T'ien, J. S., "Near-Limit Flame Spread Over a Thin Solid Fuel in Microgravity", Twenty-Second Symposium (International) on Combustion, The Combustion Institute, in press, 1988.

Time-Dependent Computational Studies of Flames in Microgravity

Naval Research Laboratory
Dr. Elaine S. Oran
Dr. K. Kailasanath
(NASA Contact: H. Ross, LeRC)
January 1987 - December 1987

Objectives: The objective of this research is to investigate fundamental problems in the combustion of premixed gases such as the ignition, propagation and extinction of gas-phase flames in zero and normal gravity.

Research Task Description: The approach we have adopted is to use detailed time-dependent, one- and two-dimensional numerical models to calculate flame properties. These models solve the multispecies coupled partial differential reactive flow equations. These models include detailed chemical kinetics mechanisms, algorithms for thermal conduction, molecular and thermal diffusion, and convective transport and include the effects of gravity.

Progress to Date: This year, the primary emphasis of our work is on studying the structure and propagation of flames near the flammability limit and to assess the effects of gravity on this structure and dynamics. We first used the two-dimensional model to verify that the predictions of the model are in agreement with experimental observations in both lean and rich hydrogen-oxygen-nitrogen mixtures. Now we are using the model to systematically study mechanisms that can lead to cellular instability. Our simulations indicate that preferential diffusion of species alone is not sufficient to cause cellular instability and suggest that a thermo-diffusive instability mechanism is the probable cause of cellular instability in lean hydrogen-oxygen-nitrogen mixtures. Currently we are studying the effects of the diffusion of intermediate species on cellular instability.

We hav also studied the effects of gravity on flame structure by comparing simulations of zero-gravity flames to upward- and downware-propagating flames. These simulations show that the effects of gravity become greater as the lean flammability limit is approached. For example, in a 1.5:1:10/hydrogen-oxygen-nitrogen mixture, gravity plays only a secondary role in determining the multi-dimensional structure of the flame. However, in a 1:1:10 mixture, an upward-propagating flame exhibits a highly curved structure which evolves into a bubble rising upwards in the tube. The zero-gravity flame shows a cellular structure. The structure of the downward-propagating flame oscillates in time exhibiting both concave and convex curvatures towards the unburnt mixture. These observations have been explained on the basis of an interaction between the buoyancy-induced Rayleigh-Taylor instability andte cellular instability. The simulations also suggest that celluiar instability grows more rapidly than Rayleigh-Taylor instability.

Publications

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Patnaik, G., Kailasanath, K., Laskey, K. J., and Oran, E. S., "Detailed Numerical Simulations of Cellular Flames," in *Proceedings of 22nd International Symposium on Combustion*, Seattle, August 1988 (in press).

Presentations

Patnaik, G., Kailasanath, K., Laskey, K. J., and Oran, E. S., "Numerical Simulations of Cellular Structure in Hydrogen Flames," *3rd Workshop on Numerical Moethods in Laminar Flame Propagation*, Leeds, England, April 1988.

Kailasanath, K., Patnaik, G., and Oran, E. S., "Effect of Gravity on Multi-dimensional Laminar Premixed Flame Structure," *39th IAF Congress*, Bangalore, India, October 1988, IAF Paper 88-354.

A Fundamental Study of Smoldering with Emphasis on Experimental Design for Zero-G

University of California at Berkeley
Professor Patrick J. Pagni
Professor A. Carlos Fernandez-Pello
Elizabeth Cantwell
Jennifer L. Newhall
NAG3-443
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Objectives: The objective of the overall research program is the design and performance of smolder combustion experiments under micro-gravity conditions. The experiments will help to understand the mechanisms controlling smoldering, and in turn the prevention and control of smolder originated fires in normal gravity and in space-based experiments. The specific objectives are: to develop theoretical models to predict the controlling mechanisms and leading non-dimensional parameters of smolder combustion; to develop ground-based experiments to determine the effect of gravity on the different modes of smoldering; to provide a data base for verification of the theoretical models. To perform drop-tower tests to obtain data on the smolder transition processes of ignition, flaming and extinction; and to use the experimental and theoretical data to design a spaced-based smoldering combustion experiment.

Research Task Description: The research program includes complementary experimental and theoretical tasks. The latter consists on the development of models of forced flow co-current and counter-current smoldering combustion, and of the transition from smoldering to flaming. The objective of the models is the identification of the smoldering non-dimensional controlling parameter and the prediction of the smolder velocity, and temperature and species distribution as a function of the fuel and flow properties. The experimental tasks consist on the determination of the effect of gravity and buoyancy, on the smolder process and its transition to flaming, for both co-current and counter-current smoldering. In the experiments, buoyancy is controlled by varying either the gravity or the gas density. The former is obtained by performing tests at the NASA Le RC, 2-2 secs drop tower, in a parabolic trajectory flight or in a space based facility. The gas density is varied by means of the ambient pressure at which the experiments are performed. The results of the experiments are used to determine the range of conditions at which smoldering is affected by gravity and to obtain information toward the design of a space based smolder combustion experiment.

Progress to Date: Research carried out during this reporting period includes experiments of the effect of ambient pressure (buoyancy) and oxidizer flow rate on the co-current smolder process, and the theoretical analysis of the transition from smoldering to flaming. The experiments using process alpha-cellulose as fuel have been completed. The measurements of the smolder velocity as a function of the oxidizer mass flow rate and pressure show that buoyancy only affects the smoldering of the alpha-cellulose (void fraction 0.85) at low air mass fluxes. At large air mass fluxes the smolder velocity is linearly proportional to the air mass flux and weakly dependent on pressure. These results are strongly dependent on the porous fuel void fraction, and a new series of tests are being carried out with open cell polyurethane foam to study how the fuel type and void fraction influences the effect of buoyancy on smoldering. Concurrent with the above experiments, a theoretical study is being carried out of the transition from smoldering to flaming. Since this process is basically a gas phase ignition mechanism induced by the hot smoldering surface, in the analysis we are using techniques already established in the study of gas ignition by hot surfaces. The method

of matched asymptotic expansions for large activation energy is being applied to determine the critical conditions at which transition from smoldering to flaming will occur at normal and micro-gravity conditions.

Publications

Dosanjh, S. and Pagni, P.J., "Forced Countercurrent Smoldering Combustion," *Second AIME-JSME Thermal Engineering Joint Conference*, Honolulu, HI, March 1987.

Newhall, J.L., Fernandez-Pello, A.C. and Pagni, P.J., "Experimental Observations of the Effect of Buoyancy on Co-Current Smoldering," *1988 Spring Meeting, Western States Section/The Combustion Institute*, Salt Lake City, UT, March 1988.

Mechanisms of Combustion Limits in Premixed Gas Flames at Microgravity

Princeton University
Professor Paul D. Ronney

Objective: The objective of this research is to study the mechanisms responsible for limit phenomena in premixed gas combustion processes. By employing microgravity experiments it is possible to eliminate the complicating and sometimes ambiguous influences of buoyant convection on these phenomena.

Research Task Description: Three types of limits have been identified which may be influenced by buoyant convection: flammability limits, stability limits, and ignition limits. A flammability limit is a limiting *composition* (e.g. most fuel-lean, most fuel-rich, or most diluted) which can sustain flame propagation in a given environment. An ignition limit is a critical *initiation condition* (e.g. a minimum energy) for the development of a self-propagating flame in a gas of a given composition. Stability limits represent critical conditions (e.g. composition, gravity level) for maintainability of a flame in a given configuration (e.g. as a plane wave, attached to a burner surface).

Besides buoyant convection, the effects of several other processes on these limit phenomena are being examined: radiant heat losses, unequal rates of diffusion of thermal energy and mass (the *Lewis number* effect), flame chemistry, conductive heat losses, and flame front curvature. The first three processes are being studied by performing experiment in a variety of fuels and oxidants in diluent gases having a wide range of radiative, diffusive, and chemical properties. The last two processes are being studied by performing experiments in two different apparatuses: a large combustion chamber with ignition at the center and cylindrical tubes of varying diameter with ignition at one end. Where theoretical understanding of these effects is lacking, new analytical models are being developed and supplemented by numerical computations.

*Progress to Date**: The results to date indicate that radiant heat losses, *Lewis number*, and flame front curvature are the dominant factors in limit phenomena at microgravity. The characteristics of all three types of limits discussed above are found to be very different in micro-g than in one-g. Two very unusual types of flame structures, not observed in one-g, have been found at micro-g. The first is called a Self-Extinguishing Flame (SEF), which is an unstable spherically expanding flame which has remarkable memory of its ignition source characteristics. SEFs are found only in mixtures with Le < 1 which are just outside the flammability limits. The second structure is a non-propagating, apparently stable spherical structure called a "flame bubble". Flame bubbles are rather remarkable in that they are apparently the only gaseous flame structures in which *convective transparent plays no role whatsoever*. These are found only in mixtures with very low Le, e.g. lean H_2-air, which are well outside the conventionally defined flammability limits.

To date experiments have been performed in the 2.2-sec. Zero-Gravity Facility at NASA-Lewis, however, a few experiments in the 5-sec. Zero-Gravity Facility and/or NASA Learjet are planned. Current instrumentation consists of direct photography and temperature measurements from thermocouples. We are planning to develop a more advanced flame front imaging system based on schlieren, IR, and UV techniques and non-intrusive temperature and chemical species concentration diagnostics based on an optical spectroscopic technique.

The results of these micro-g experiments have applications to the assessment of fire safety in spacecraft. The understanding of limit mechanisms gained from this work may also lead to improved fire safety in terrestrial environments where gaseous fires and explosions may occur, e.g. oil refineries, chemical processing plants, and mine shafts. This understanding is also relevant to the development of cleaner burning and more fuel-efficient energy conversion systems through the use of very fuel-lean combustible mixtures.

*Professor Ronney has previously done work in the subject area of this grant and thus there are some results to report.

The Effects of Gravity on Flame Spread Involving Liquid Fuel Pools

NASA Lewis Research Center
Dr. Howard Ross
In-House

Objectives: The objective of the study is to increase the fundamental understanding of flame spread involving liquid fuel pools through experiments in a microgravity environment. Gravity affects the liquid fuel and gas phase motions and as such influences the supply of oxidizer and heat transfer ahead of the flame. Its role is sufficiently complex so that it is not clear a priority whether the flame spread rate will be faster or slower in reduced gravity than in normal gravity. To improve the understanding of the role of gravity, a range of liquid Grashof numbers on the order of 0.01 to 1,000,000 will be studied.

Research Task Description: The first task of the research program is to determine if a quiescent, flat liquid-gas interface can be achieved in a time frame compatible with ground-based reduced-gravity test facilities. Toward this end, a drop rig will be constructed to develop and test various container designs and shapes.

If successful in "pinning the interface", an apparatus will be designed and constructed for use in a pressure vessel for reduced gravity tests to be performed in the 5 second facility at LeRC. It is planned to study the flame spread rate dependence on gravity, pressure, container material and dimension, and oxidizer concentration for fuels above and below their flash points at normal room temperature (ethanol, methanol, propanol, and butanol). Some normal gravity tests will be performed to establish baseline and additional scientific data. A comparison of experimental results to existing numerical models will then be made. These models may be modified as needed.

Depending on the results of the efforts, the justification and feasibility of proceeding to a space experiment or of further ground-based microgravity research will be determined.

Progress to Date: The drop rig for liquid-gas interface testing was designed, constructed, and operated for about 25 drops. Tests have shown it is possible to reach the desired conditions within 2.2 seconds for shallow (< 1 mm) pools of methanol in 15 cm diameter trays. Deeper pools are also "pinned" successfully, but the motion resultant from the transition from normal to reduced gravity is not damped completely within the time frame. All indications are that they would become quiescent given more reduced gravity time. Tests in the 5 second facility at LeRC further verified these indications.

Hardware was designed and constructed to begin the flame spread testing in the 5 second facility. Initial checkout of the safety features (e.g. trapdoor to quench fire, nitrogen purge system, and venting to vacuum) were completed. Photography and ignition systems are currently being finalized.

Presentations

Ross, H. D. and Pline, A., "Equilibrium Times for Liquid-Gas Interfacial Shapes Transitioning from Normal to Reduced Gravity," AIChE Annual Meeting, November 1988, Washington DC

Ignition and Flame Spread Above Liquid Fuel Pools

University of California, Irvine
Professor William A. Sirignano
F. H. Tsau
D. N. Schiller
NAG3-627
(NASA Contact: Boyd Bane, LeRC)
January 1, 1987 - March 31, 1988

Objectives: The objectives of this program are to investigate thermal and fluid dynamic behaviors of fluids within an axisymmetrical container as related to ignition and flame propagation phenomena with liquid fuel pools. The two-dimensional and thre-dimensional aspect of the flow is of particular interest. Th onset of the three dimensionality is expected above a certain critical Grashof number. Promising speculations were obtained from a two-dimensional numerical study.

Research Task Description: (1) A 3-D Cartesian numerical solver is adopted and simple 2-D cases are tested to debug the program; (2) The program is then adapted to a more general solver which is capable of doing both rectangular and cylindrical simulations; A simple case will be tested; (3) The resulting solver will be used to study the target problem; (4) Parameter studies will continue with two-dimensional code with comparison to experimental results from NASA Lewis Research Center; and (5) Experiments will be designed for reduced gravity facilities in collaboration with NASA Lewis.

Progress to Date: Step 1 was completed successfully. Steps 2, 4, and 5 are in progress.

Presentations

Ross, H. D., Schiller, D. N., and Kim, H., "Behavior in Normal and Reduced Gravity of an Enclosed Liquid/Gas System with Nonuniform Heating from Above," to be presented at 27th Aerospace Sciences Meeting, Reno, Nevada, January 1989.

7. EXPERIMENTAL TECHNOLOGY AND GENERAL STUDIES

Modeling Directional Solidification Furnaces/Processes

NASA Lewis Research Center
Dr. Arnon Chait
In-House

Objectives: The underlying objective ofthis study is to develop a generic numerical tool for examining the interaction between the furnace and sample transport phenomena in directional solidification processes. A second goal is to implement an optimization procedure into the model to allow the modification of furnace components and/or operating parameters in order to achieve a user-designated design criterion (e.g. a prescribed solidification interface). The study will concentrate on flight furnaces and will provide a means for experimenters to model the interaction of specific hardware with their materials.

Research Task Description: A generic finite-element based code is chosen for computing the thermofluid transport phenomena in the furnace/sample. Specific enhancements to the code may include the following additions:

- heat of fusion using either a fixed grid (enthalpy) or a moving grid approach
- two solute transport equations
- appropriate temperature and mass boundary conditions at the melt/solid interface
- gray body radiation scheme
- furnace simplification algorithms (boundary curvature method)
- optimization loop to allow modification of user-selected regions (e.g. furnace components) and/or operating parameters as to optimize a designated criterion
- database development for flight hardware.

The design tool will reside in a graphics workstation environment, complete with user interface and remote processing capabilities.

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Electrostatic Containerless Processing Technology

Jet Propulsion Laboratory
Daniel D. Elleman
Won-Kyu Rhim

Objectives: The primary objective of the task is the development of the science and technology base required for containerless positioning and manipulation of various materials using electrostatic and electrophoretic forces. Experimental and theoretical investigations will be carried out so as to develop the following system. A focused radiator furnace which will operate either in a vacuum environment or in a controlled gaseous environment that will provide for heating and melting samples up to 2000 C. This system would also provide for rapid quenching of the sample in a containerless manner. A method of measuring certain physical parameters such as surface tension and viscosity will be developed for this type of positioning device. It has been demonstrated that this type of system can position either metallic or nonmetallic samples as well as liquid or solid samples.

Research Task Description: This task is engaged in developing various electrostatic and electrophoretic containerless positioning devices. In addition a hybrid electrostatic acoustic positioning module has been developed that operates at ambient temperatures and has been used to grow a variety of protein crystals from solution. In addition this system has been used to investigate undercooling of samples at or near ambient temperatures. The high temperature vacuum focused radiator levitator system is presently capable of heating metallic samples up to 1000°C and measurement of electrical charge exchange between the sample and the electrodes have been made with the system. Small low density samples have been levitated in the laboratories one g environment with both the pure electrostatic and electrostatic acoustic hybrid levitators. Both conducting samples such as metallic shells and nonconducting samples such as glass shells have been successfully levitated demonstrating that the technique is sample independent. The electrostatic module has been tested in a low gravity environment provided by the KC 135 and large one centimeter diameter samples were positioned and controlled successfully.

Progress to Date: A series of fluid dynamic experiments were conducted with the electrostatic acoustic hybrid positioning system to investigate the dynamic stability and equilibrium shapes of charged rotating liquid drops. Drops 3 mm dia. of water and water glycerin mixtures were levitated electrostatically and rotated with an acoustic torque. Families of axisymmetric shapes, two-looped shapes and eventual fissioning of the drop were observed. The results seem to agree well with the theory for uncharged drop developed by Brown and Sereven.

Charge exchange between a metallic sample and the electrodes has been measured while the sample is heated up to 1000°C by a focused radiator lamp. Measurements show that photo electrons are emitted by the sample from the UV light from the lamp. If the UV is suppressed by a filter, the photoelectron emission is reduced and accumulation of large negative charge is observed at high temperatures. The charge loss or gain is greatly dependent on the cleanliness of the sample as well as the temperature.

Publications

Rhim, W-K, Chung, S. K., and Elleman, D. D., "Equilibrium Shapes and Stability of Rotating Charged Drops," *Proceedings of First National Fluid Dynamics Congress*, 1988 (in press).

Rhim, W-K, Chung, S. K., and Elleman, D. D., "Experiments on Rotating Charged Drops," *Proceedings of Third International Colloquium on Drops and Bubbles*, Monterey, September 1988 (in press).

Advanced Containerless Processing Technology

Jet Propulsion Laboratory
Dr. Taylor G. Wang
In-Center
Continuing Task

Objectives: The objectives of this task are to: (1) develope next generation systems for Space Station containerless experiments such as electromagnetic-acoustic hybrid system, electrostatic-acoustic system, and high temperature acoustic system; (2) advance the knowledge base of high intensity and high temperature acoustics in the area of non-linear, instability, and streaming; (3) investigate the effects of acoustic electrostatic, electromagnetic, and temperature fields on the levitated sample, and vice versa; and (4) provide the assistance to develop a set of high temperature materials experiments for Space Station.

Research Task Description: Many of the materials research experiments to be conducted in the microgravity science and applications program require manipulation and control of weightless molten materials in a non-contaminating method. In these experiments, the melt is positioned and formed withina container without physically contacting the container's wall. An acoustic method which was developed at JPL has demonstrated the capabilities of positioning and manipulating a moderate temperature sample. This has been accomplished in an earth-based laboratory and in a zero-g environment of short duration. However, many important facets of high temperature containerless processing technology have not been established yet, nor can they be established from the room temperature studies, because thesetails of the interaction between an acoustic field and a molten sample are largely unknown. For example, a molten sample positioned at the middle of the acoustic fields is continually under the influence of acoustic fields. The effect of these fields on the nucleation and solidification processes can only be determined by actual testing.

Publications

Lee, C. P., and Wang, T. G., "Acoustic Radiation Potential on a Small Sphere Due to Two Orthogonal Standing Waves," J. Acoust. Soc. Am. 83, 2459 (1988).

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B. FLIGHT EXPERIMENTS

1. ELECTRONIC MATERIALS

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Compound Semiconductor Growth in Space

NASA Langley Research Center
Dr. A. L. Fripp
W. J. Debnam
I. O. Clark
Dr. R. K. Crouch, NASA Headquarters
In-House

Objectives: The objectives of this research is to determine the effects of gravity driven convection on the growth parameters and crystal properties of compound semiconductors.

Research Task Description: The Microgravity Science effort at the Langley Research Center is centered on the growth of the compound semiconductor alloy, lead tin telluride. Lead tin telluride is a substitutional alloy of lead telluride and tin telluride that is miscible over the entire compositional range. The semiconductor properties of this material are dependent on the ratio of the two components and consequently, the uniformity of an array of devices is dependent on good compositional control.

This material was chosen for microgravity research for a number of reasons. Lead tin telluride is not only a useful semiconductor material which has been used for construction of infrared detectors and tunable diode lasers but it also has a similar phase diagram to other compound semiconductors of interest such as mercury cadmium telluride and mercury zinc telluride. Lead tin teliuride is very interesting from a purely scientific point of view in that it is both solutially and thermally unstable, but in a one dimensional analysis with growth axis parallel to the gravity vector, only one instability works, per orientation, at a time. This double convective instability cannot be made stable by balancing thermal and solutal expansion in a high temperature gradient. Lead tin telluride is amenable to study for it is easily compounded; it has a relatively low vapor pressure; it is single phase and there is existing, though limited, literature on its growth and properties.

The desired growth mode is of course one in which convection is zero so compositional steady state can be reached. However fluid dynamic calculations have shown that finite convection exists in the physical configuration used in crystal growth experiments even at $1xE^{-8}$ Earth gravity if there is a density gradient orthogonal to the gravity vector. However, due to the residual atmosphere the minimum gravity level expected on the Space Shuttle is roughly the order of $1xE^{-7}$ Earth gravity. Hence experiments are designed such that interface movement i.e., growth rate, is greater that the anticipated fluid velocity.

Other work at Langley ranges from fundamental studies involving the direct measurement of both steady and oscillatory fluid flow in a crystal growth melt to efforts in furnace design and calibration. Also of interest is an effort to measure the melt-solid interface shape and position as this is of great importance to understanding the crystal growth process.

Progress to Date: This has been a very eventful year in gaining a greater insight into the fundamental properties of crystal growth. Our greatest achievement has been real time measurement and image enhancement of the interface shape and position during crystal growth. Another achievement has been the measurement and characterization of fluid flow in a melt system. A greater insight into the properties of the diffusion

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boundary layer has come about from the results of the STS 61A flight and ground based experiments are still in progress to extend this understanding.

Presentations

Fripp, A. L., Debnam, W. J., Woodell, G. A., Berry, R.F., Barber, P.G., Huang, Y., Simchick R., and Carlson, F. M., "Interface Movement Related to Growth Rate in Bridgman Crystal Growth"; ; AIAA 26th Aerospace Sciences Meeting, Reno, Nevada; January 11-14,1988.

Barber, P. G., Fripp, A. L., Debnam, W. J., Berry, R. F., Stacy, K., Huang, Y., and Simchick, R. T., "Lower Limits of Detection of Interface Properties," American Conference on Crystal Growth-West, Stanford Serria Camp, Fallen Leaf Lake, CA, June 6-10, 1988.

Knuteson, D. J., Narayanan, R., Debnam, W. J., and Fripp, A. L., "Thermal Study of Convection in Low Prandtl Number Liquids," American Conference on Crystal Growth-West, Stanford Serria Camp, Fallen Leaf Lake, CA, June 6-10, 1988.

Woodell, G. A., Fripp, A. L., and Knuteson, D. J., "Calculation of Growth Rates for a Directionally Solified Semiconductor Crystal," Asyst'88 International Conference, University of Rochester, Rochester, N.Y., October 9-11, 1988.

Woodell, G. A., "Data Acquisition From a Crystal Growth Furnace," Asyst'88 International Conference, University of Rochester, Rochester, N.Y., October 9-11, 1988.

Woodell, G. A. and Knuteson, D. J., "Data Acquisition for the Thermal Study of Convection in a Liquid Metal During Crystal Growth," Asyst'88 International Conference, University of Rochester, Rochester, N.Y., October 9-11, 1988.

Barber, P. G., Fripp, A. L., Debnam, W. J., Berry, R. F., Stacy, K., Huang, Y., and Simchick, R. T. "X-Ray Intensifiers and Image Enhancement to Determine the Location and Shape of Melt-Solid Interfaces in Bridgman Growth of Compound Semiconductors," American Conference on Crystal Growth-East, Atlantic City, NJ, October 18-21, 1988.

Huang, Y., Debnam, W. J., and Fripp, A. L., "Interface Shape During Vertical Bridgman Growth of PbSnTe," American Conference on Crystal Growth-East, Atlantic City, NJ, October 18-21, 1988.

Hurst, J. H., Anderson, T. J., Fripp, A. L., and Debnam, W. J., "Electrochemical Visualization of Convection in Liquid Metal," American Conference on Crystal Growth-East, Atlantic City, NJ, October 18-21, 1988.

Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology
Professor Harry C. Gatos
Dr. Jacek Lagowski
NSG 7331
(NASA Contact: Dr. Roger Crouch HQ)
December 1, 1987 - November 30, 1988

Objectives: The objectives of the research are to investigate defect engineering in zero gravity environment as a means for achieving GaAs single crystals with device properties and processing characteristics surpassing theoretical limits of perfect bulk GaAs single crystals.

Research Task Description: The research is aimed at developing defect control and characterization techniques for achieving quantitative relationships among conventional (growth rate, therma! gradients, heat and mass flow) and new (stoichiometry and postsolidification cooling rate) crystal growth parameters --electronic properties of GaAs and device processing characteristics. Zero gravity is treated as an environment which offers enhanced controllability of defects and this can be utilized for engineering GasAs crystals with new properties resulting from the beneficial role of native defects and theor complexes. This approach is expected to point the way to achieving GaAs crystals of other compound semiconductors with device properties surpassing theoretical quality of the perfect (defect free) crystals..

Progress to Date: During the past year our research was focused on establishing a fundamental underpinning for effective defect engineering in GaAs. Toward that goal we have refined certain characterization techniques: high resolution optical absorption and low temperature scanning photoluminescence. The understanding of postsolidification cooling of GaAs and its relationships with device processing characteristics has been significantly advanced. We have also continued GaAs crystal growth experiments in a triangular prism configuration with emphasis on the effects of surface tension on the growth morphology.

Publications

Gatos, H. C. and Lagowski, J., "Comment on 'Identification of a Defect in a Semiconductor: EL2 in GaAs'," Phys. Rev. B 26, 7668 (1987).

Bugajski, M., Ko, K. H., Lagowski, J., and Gatos, H. C., "Native Acceptor Levels in Ga-Rich GaAs," Appl. Phys., 1988 (in press).

Presentations

Skowronski, M., Lagowski, J., Milshtein, M., Kang, C. H., Dabkowski, F., and Gatos, H. C., "The Effects of Plastic Deformation on Electrical Properties of GaAs," 14th International Conference on GaAs and Related Compounds, Institute of Physics Conference Series, 1988.

Gatos, H. C., Lagowski, J., and Wu, Y., "Growth of GaAs from a Free Surface Melt under Controlled Arsenic Pressure in a Partially Confined Configuration," 39th IAF Congress, Bangalore, India, October 1988.

A Comparative Study of the Influence of Convection on GaAs

GTE Laboratories, Inc.
Dr. James A. Kafalas
Ben Yacobi
Alfred Bellows
David Matthiesen
Brian Ditchek
NAS3-24644
(NASA Contact: Dr. R. Lauver, LeRC)
September 6, 1985 - September 5, 1989

Objectives: The objective of this study is to determine the effects of buoyancy driven fluid flow on the properties of melt grown GaAs crystals.

Research Task Description: Baseline GaAs crystals grown in the convection-free environment of the Space Shuttle will be compared to crystals grown on earth under various fluid flow conditions as determined by gradient orientation and the presence and orientation of a magnetic field. The characterization of the GaAs crystals will correlate the degree and nature of the convection with macro- and microsegregation effects, dislocation density distribution and electronic properties. The data will be interpreted based on model calculations of the fluid flow patterns in the melt under the various growth conditions. The improved understanding of the role of convection in the growth of GaAs gained from the proposed research will contribute to the refinement of GaAs growth techniques to produce substrate material with improved homogeneity and lower dislocation densities.

Solution Crystal Growth In Low-g

Alabama A&M University
Dr. R. B. Lal
Dr. W. R. Wilcox, Clarkson University
Dr. J. D. Trolinger, Metro Laser
NAS8-36634 (NASA Contact: Mr. Rudolph Ruff, MSFC)
September 25, 1986 - December 31, 1988

Objectives: The objectives of this research project are: 1) to grow crystals to triglycine sulfate (TGS) using polyhedral seeds using modified Fluid Experiment System (FES); 2) to study holographic interferometry tomography of the fluid fields in three dimensions and, 3) to study the fluid holography of tracers, and to estimate the influence of g-jitter on the growth rate.

Research Task Description: This project involves a reflight of an earlier experiment, "Solution Growth of Crystals in Zero-g," flown on Spacelab-3 mission. Single crystals of TGS will be grown in the modified FES using (001) oriented polyhedral seeds. Experiments are underway to determine the proper seed size, so that natural (001) face seeds can be used for the flight experiment. Also, experiments are conducted in the Ground Control Experiments Laboratory (GCEL) where a test cell similar to flight is being used with holography system. The optical part of the FES system has been mocked up including the FES crystal growth cell and holography system. Experiments have been conducted to determine the size, type, and number density of particles that should be used in the FES to monitor convective flow. During GCEL runs holograms are recorded with particles 50μ and 100μ. Proper design of holographic optical element (HOE) has to be determined. The HOE will split the incming beam at the first window into three beams at different angles and the second HOE on the other window will reconverge the three beams so that they all hit the hologram. This will provide three independent views of the fluid field. Th grown crystals will be characterized for electrical properties and defects and I.R. detectors will be fabricated.

Progress to Date: The crystal orientation and size has been determined by laboratory experiments and also experiments in GCEL. The design of the HOE has been completed. The particle size of 50μ or 100μ is found to be adequate to be resolved with the resolution of FES optics system. Further experiments in GCEL are planned to resolve the number density of particles using holography. Preliminary test with HOE has been completed. Actual spare optical windows of FES will be coated with HOE's and tested in the GCEL.

Growth of Solid Solution Crystals

Marshall Space Flight Center
Dr. S. L. Lehoczky
Dr. F. R. Szofran
In-House
Continuing task

The major objective of this research is to establish the limitations imposed by gravity during growth on the quality of bulk solid solution semiconducting crystals. An important goal is to explore the possible advantages of growth in the absence of gravity. The alloy system being investigated is $Hg_{1-x}Cd_xTe$ with x-values appropriate for infrared detector applications in the 8 to 14 m wavelength region. Both melt and Te-solvent growth are being considered. The study consists of an extensive ground-based experimental and theoretical research effort required to define the optimum experimental parameters fo the planned flight experiments. $Hg_{1-x}Cd_xTe$ is representative of several II-VI alloys which have electrical and optical properties that can be compositionally tuned to meet a wide range of technological applications in the areas of sensors and lasers with applications to opticl computing and communications as well as the national defense.

A series of $Hg_{1-x}Cd_xTe$ alloy ingots ($0<x\leq0.6$) has been grown from pseudobinary melts by a vertical Bridgman-Stockbarger method using a wide range of growth rates and thermal conditions. Precision measurements were performed on the ingots to establish compositional distributions for the ingots. Growth rates and thermal conditions required to obtain the desired growth interface shape have been established for the system.

To assist the interpretation of the results and the selection of optimum in-flight growth parameters, the pseudobinary phase diagram ($0\leq x\leq1$), liquid and thermal diffusivities ($0\leq x\leq0.3$), and the specific volumes as a function of temperature ($0\leq x\leq0.15$) have been measured. From these measurements and other available data, the heat capacity, enthalpy of mixing, and the thermal conductivity of pseudobinary melts have been calculated using a regular associates solution model for the liquid phase. A one-dimensional diffusion model that treats the variation of the interface temperature, interface segregation coefficient, and growth velocity has been used to establish effective diffusion constants for the alloy system. Theoretical models have been developed for the temperature distribution and the axial and radial compositional redistribution during directional solidification of the alloys. These models are sufficiently accurate that they will be used alog with te experimental results to select parameters for the first flight experiment.

A microscopic model for the calculation of charge-carrier concentration, Fermi energy, and conduction-electron mobility as functions of x, temperature, and both ionized and neutral defect densities has been developed. For selected samples, measurements were performed of electron concentration and mobility from 10-300K. The experimental data were in reasonably good agreement with theory and were successfully analyzed to obtain donor and acceptor concentrations for various processing conditions.

The crystal growth studies have been extended to include additional Hg-based II-VI alloys. Large crystal ingots of HgZnTe and HgZnSe have been successfully grown by the Bridgman-Stockbarger method and a detailed theoretical analysis of the measured

axial compositional distribution in the ingots was used to establish for the first time effective HgTe-ZnTe and HgSe-ZnSe interdiffusion coefficients for the molten alloys. Both the Te and Se-based alloys showed improvements in lattice strength resulting from the substitution of Zn into the respective Cd lattice sites. In particular, measured electrical and optical properties of the HgZnSe crystals indicated that the addition of Zn to the HgSe system was effective in stabilizing the electrical properties, thus providing the first direct experimental confirmation for predicted improvement in lattice stability against point-defect formation resulting from Zn-additions.

Microhardness measurements were performed on selected waffers cut from the various alloy crystals. For each alloy, measurements were made for several alloy compositions. The microhardness results support the above conclusions. Sample cartridges needed for the integration of the flight HgCdTe alloy samples into the space flight hardware being developed.

Publications

Lehoczky, S. L., Szofran, F. R., Su, C-H., Cobb, S., and Andrews, R. N., "Crystal Growth of Solid Solution Systems by Directional Solidification," in *Proceedings of ASM International '87 Materials Congress*, 1988, in press.

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Vapor Crystal Growth of Mercuric Iodide

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January 1988 - January 1989

Objectives: Single crystals of mercuric iodide are used in high-efficiency x-ray and gamma ray detectors operating at ambient temperature. Optimal operation of the devices is determined to a large degree by the density of structural defects in the single crystalline material. Since there are strong indications that the quality of the material is degraded by the effects of gravity during the growth process, a program was initiated to grow one or more crystals of mercuric iodide in the reduced gravity environment of space.

Specifically, there are two reasons to perform the space experiments:

1. Single crystals of mercuric iodide are prone to slippage under the effect of gravity, especially at the elevated growth temperatures, with a concurrent decrease in structural quality.

2. It is not clear what effects convection flows in the vapor phase have on the growth and the homogeneity of the crystals. Growth in reduced gravity would provide information regarding these questions.

Research Task Description: The first experiment, performed during the flight of Spacelab 3 (April 29-May 6, 1985), was highly successful in the sense that all scientific objectives were fulfilled. The structure of the space-grown crystal was more homogeneous and the critical electronic properties were increased by a factor of seven compared with the best earth-grown crystals.

Preparations are underway for the next experiment, to be flown on the first flight of the International Microgravity Laboratory (IML). Present ground-based research and experimental development activities concentrate on improving the control system of the flight equipment and increasing the temperature of the growth process so that larger crystals can be obtained in the limited time available during the flight. The ground based research will be extended in the near future to include extensive measurements of the temperature profile in the growth ampoules, so that more detailed computations can be made of the convection patterns in the vapor phase.

Progress to Date: Discussions have taken place with the NASA Sensor Working Group to use high-quality mercuric iodide crystas in advanced systems for planetary exploration and astrophysics experiments.

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Vapor Growth of Alloy-Type Semiconductor Crystals

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March 1978 - October 1989

Objectives: The present effort is part of a continuing research program directed towards the investigation of basic vapor transport phenomena and of crystal growth properties of electronic materials. The primary objectives of ground-based studies are the development and definition of optimum experimental parameters for flight experiments. The specific experiments to be performed in a microgravity environment include the investigation of vapor transport and crystal growth phenomena of the $Hg_{1-x}Cd_xTe$-HgI_2 system. Emphasis for this system is on the mass flux, on the unseeded growth of bulk crystals, and on the growth of epitaxial layers. The above experiments are performed in closed, fused silica ampoules.

The objectives of the $Hg_{1-x}Cd_xTe$ experiments are to determine the positive effects of microgravity on vapor phase crystal growth of ternary, alloy-type materials in terms of chemical and structural microhomogeneity, and of electrical properties of crystals grown in space. In order to achieve these goals it is necessary to perform corresponding crystal growth studies on ground which will serve as a comparative basis for the meaningful analysis of microgravity effects. Thus, the objectives of ground-based studies are to determine the effects of gravity-driven convection on mass transport rates on on crystal morphology for the bulk and epitaxial growth of $Hg_{1-x}Cd_xTe$ crystals. These objectives include the thermodynamic and fluid dynamic analysis of the mass transport processes, and the characterization of grown crystals in terms of defects and of electrical properties. The ultimate goal is the quantitative correlation between crystal morphology, electrical properties, and growth parameters for this system.

Research Task Description: Earlier studies demonstrated the effects of gravity-driven convection on mass flux and morphology of $Hg_{1-x}Cd_xTe$ bulk crystals. The continued experimental tasks are directed towards the investigation of the effects of temperature fluctuations and of the geometry of the condensation region of the ampoule on mass flux and crystal morphology. In addition, the effects of aspect ratio of the ampoule on the mass transport and crystal growth properties will be investigated. The ground based tasks include the investigation of the above effects for different orientations of the density gradient with respwect to the gravity vector, and for different transport agent (HgI_2) pressures and temperature profiles. The crystal characterization techniques include various microscopic and x-ray diffraction methods, chemical etching, and electrical measurements. The ground-based studies are designed to optimize experimental parameters for future flight experiments.

The major tasks of ground-based studies of the seeded growth of $Hg_{1-x}Cd_xTe$ layers by chemical vapor transport reactions involve systematic investigations of the growth rate, morphology, homogeneity, and electrical properties of HgCdTe layers. These studies include measurements of the effects of substrate orientation relative to the density gradient, of temperature profile effects, and of transport agent pressure on the above properties. They are performed under horizontal and vertical stabilizing conditions with the goal to observe the effects of convective interferences on layer morphology and properties. The results of on-going ground-based studies are

continuously evaluated and are used for the systematic modification of grown parameters with the important goal to define optimum experimental conditions for the microgravity experiments of this system.

In addition to the experimental tasks, theoretical efforts involve the quantitative thermodynamic analysis of the systems under investigation, the computation of fluid dynamic parameters, and the consideration of other possible effects on fluid flow under vertical, stabilizing and microgravity conditions. An important aspect of the theoretical effort is the further development and improvement of transport models for diffusion limited mass transport of simple and of multi-component, multi-reaction vapor transport systems.

Progress to Date: Progress accomplished earlier under this program includes mass flux and crystal growth properties of bulk and layer-type crystals. Dynamic microbalance techniques were employed, for the first time, to determine quantitatively the vapor pressure of Hg over $Hg_{1-x}Cd_xTe$ for different compositions within the homogeneity range. This work led to the direct in-situ determination of the Hg vacancy formation for this important material. These results provide valuable information for the further elucidation of the mechanism of vacancy formation of $Hg_{1-x}Cd_xTe$. Our recently developed thermodynamic model of the $Hg_{0.8}Cd_{0.2}Te$-iodine transport system has been extended to predict the composition of the transported crystals for different pressures and temperatures. Considering the complexity of the $Hg_{1-x}Cd_xTe$-HgI_2 transport system, the theoretically poredicted compositions are in good agreement with experimental observations for a wide range of compositions. This computational model is expected to be generally applicable for the prediction of mass transport properties in terms of mass flux and overall composition of transport products for the diffusion dominated vapor transport of ternary alloy-type materials. Such a predictive capability is of practical significance for the design, performance, and evaluation of crystal growth experiments on earth and in microgravity environment.

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2. SOLIDIFICATION OF METALS, ALLOYS AND COMPOSITES

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Alloy Undercooling Experiments in Microgravity Environment

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March 1, 1988 - February 28, 1989

Objectives: The objectives of this research are: (1) to evaluate containerless melting and solidification of nickel and iron base alloys with and without softened glass coatings, (2) to develop techniques for the study of recalescence and growth behavior during solidification of undercooled alloy melts at zero gravity, (3) to develop an understanding of undercooling phenomena in microgravity, and (4) to develop an understanding of the microstructures so produced.

The scientific aim of the current and proposed modeling studies and ground-based experiments is to obtain baseline data and a baseline understanding of nucleation, growth, solidification, and structure of highly undercooled alloys on which comparable experiments will be conducted in space. Concomitantly, experimental techniques are being developed for performing and analyzing undercooling experiments in space and on the ground.

Research Task Description: The first alloy undercooling experiment in a microgravity environment was performed during the Columbia STS 61-C mission in January 1986. The results of the flight experiment have been published. The directly related ground-based experimental and analytical studies include thermal history measurement during the rapid recalescence and solidification of undercooled alloys, dendrite growth rate measurements, metallographic studies, and modeling of dendritic growth in undercooled melts. These results have been reported in published papers.

Progress to Date: Recent work has been performed using Ni-Sn and Fe-Ni alloys. In the Ni-Sn system, alloys of low Sn content (1, 2, and 5% Sn) have been used in experiments to understand the effect of composition and alloy freezing range on the dendritic growth behavior of undercooled alloys. These results have been compared with earlier work on Ni-25% Sn, and show that lower solute contents and more narrow freezing ranges are associated with significantly higher dendrite growth velocities and nonequilibrium solute partitioning at high undercoolings. It has also been shown that, for alloys of moderate alloy content and freezing range (Ni-5% Sn), the solid which forms initially can be superheated during rapid recalescence and later rapidly remelt, a discovery in agreement with earlier modeling work in this program. The experimental work on the Ni-Sn system is nearing completion.

Experimental work on the Fe-Ni system has concentrated on the 5, 10, and 30% Ni alloys. Since there is a peritectic in the Fe-Ni system, there are two high-temperature crystal structures which can nucleate in undercooled melts. It has been found that in Fe-10% Ni the metastable BCC phase forms first, in preference to the stable FCC phase, when there is initial undercooling. This can be detected in bulk specimens only by high-speed measurements of the thermal history during recalescence, since the metastable phase disappears quickly and is not found in the final structure of large specimens. Metastable phases can, however, be retained in rapid solidification processes, and some modeling work on this nucleation phenomenon has been performed. Fe-Ni alloys are also being studied because of the narrow equilibrium solidification temperature range of the alloys. At high undercoolings, dendrite growth occurs at very

large velocities, and nonequilibrium solute partitioning is an important consideration.

Some initial experiments have also been performed on Fe-B alloys. These alloys are interesting because of the very low partition ratio, wide freezing range, and potential glass-forming ability. Early results indicate that at moderate undercoolings dendrite growth velocity is very low, in comparison to the alloys mentioned above. Future work will expand our understanding of the dendritic growth of alloys in which increasing viscosity and slower solute diffusion are significant.

A detailed and comprehensive model was developed for processes occurring behind the dendrite tips during recalescence and subsequent solidification. Based on the foregoing results, a full model has been developed and presented for this solidification behind the dendrite tips that takes place by thickening and branching of the primary arms. The dendrite structure which forms is very fine, with a secondary dendrite arm spacing on the order of 10 μm at the higher undercoolings. The residual interdendritic undercooling dissipates in an extremely short time, with concomitant rapid recalescence. Rapid recalescence continues until the residual liquid is no longer undercooled with respect to the solid dendrite arms. After this, recalescence continues at a much slower rate which is controlled by coarsening of the initial dendrite structure. The extensive ripening which occurs during the slow recalescence is one factor which accounts for the observation that the maximum recalescence temperature is at or near the "equilibrium temperature."

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Isothermal Dendritic Growth Experiment

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Objectives: The objective of this flight experiments is to assess the influence of gravity on the growth kinetics and solidification morphology of freely growing dendrites.

Research Task Description: The work has focussed on three areas: (1) growth chamber design and development, (2) evaluation of the photographic data collection system, and (3) data analysis and reduction procedures.

Progress to Date:

Growth Chamber Design and Development

The laboratory growth chamber has been redesigned for the flight experiment. The design of the flight growth chamber has incorporated a number of features not present in the laboratory model. The flight chamber design has passed a Critical Design Review held at NASA Lewis Research Center on April 6, 1988. Preliminary testing of engineering models of the flight chamber has shown that the design will meet the engineering and scientific requirements for flight aboard STS.

An exhaustive set of materials compatibility tests were run in order to obtain construction materials with the least interaction with SCN. These materials were found to be a subset of stainless steels. To obtain free dendritic growth, driven only by diffusion of heat through the liquid, it is necessary to induce dendritic growth at the center of the chamber. This is accomplished via a capillary injector (stinger). The stinger consists of a stainless steel tube, with a glass tip sealed to the end, in the growth chamber. The glass tip is pulled to a small diameter, approximately one millimeter outside diameter and 200 micron inside diameter. The end of the stinger outside the growth chamber is sealed with a welded stainless steel plug and capped with a copper plate.

The initiation of the growth front in the stinger is accomplished with four small thermoelectric coolers attached to the copper plate on the end of the stringer stem. The coolers are potted in epoxy to provide thermal isolation from the thermostatic bath. These coolers will allow the central microprocessor to initiate growth when all of the experimental conditions have been met. Once the coolers are switched on they drive the temperature of the stinger well below the nucleation temperature of SCN. Once initiated, the growth front then proceeds down the stinger where it emerges in the center of the growth chamber to be photographed. The growth chamber must provide two unobstructed orthogonal viewing axes. Thus, the chamber contains four windows. The windows consist of glass flats sealed to stainless steel bezels which are, in turn, electron beam welded to the chamber body. Sample volume change compensation, which is a

special problem under low gravity, also had to be included in the growth chamber design. The thermal expansion of the solid from 25 C to 58.1 C, the phase-change volume expansion, and the thermal expansion of the liquid from 58.1 to 62 C must be compensated for to prevent stray vapor cavities from forming and to prevent overpressuring the specimen chamber. In addition the system must remain hermetically sealed, prevent free surface formation such as shrinkage pores, and be constructed entirely from stainless steel and glass. The volume expansion and free surface control are accomplished with a stainless steel bellows electron-beam welded between the top and bottom halves of the growth chamber. The bellows throw and spring rate are sufficient to compensate for all volume changes with a large safety factor. The growth chamber is sealed under vacuum and the external pressure transmitted through the bellows from the thermostatic bath is sufficient to prevent any free surface formation. The final component of interest in the growth chamber is an ultra stable thermistor. This thermistor serves two functions. First, when used in conjunction with a laboratory temperature standard during ground based testing, the purity of the SCN in the chamber can be checked. Secondly, during flight, this thermistor may be used to cross-calibrate all temperature sensors located inside the thermostatic bath to the actual measured melting temperature of the sample.

Results of the preliminary tests of the engineering models follow: The chamber was capable of maintaining the SCN at an undercooling of 1.0 K for a period of an hour. The bellows maintained a void free sample and accounted for expansion through multiple thermal cycles of the chamber. The insitu thermistor indicates that melting occurs over a period of 90 minutes, with a plateau slope of 0.2 mK/min at a melting temperature of 59.5 C, which is more than adequate for melting point determination. The melting plateau tests on the engineering models indicate cleaning and filling procedures are capable of maintaining the purity of succinonitrile. Tests of the thermoelectric coolers show that the original coolers were not of sufficient power to reliably initiate dendritic growth at all supercoolings (yielding 5 degrees of cooling) and are being substituted with larger coolers which in laboratory tests are capable of achieving up to 15 degrees of cooling. Dendritic initiation was reliable at undercoolings larger than 0.5 K and the subsequent emergence of the dendrite at the tip of the stinger during multiple runs occurred within seconds of each other, demonstrating the reproducibility of dendritic growth. Additional testing will be required to optimize the performance of the thermoelectric cooling assembly.

Although further testing of the engineering models is required, several areas of the design (e.g., placement of the fill tube, window shape and size) have been identified for redesign. Also several ways to reduce weight and overall length are being considered and will be incorporated into the next models.

Photographic Data Collection System

Several photographic testing sessions have been conducted over the past years in conjunction with NASA Lewis Research Center. These sessions have helped identify and define certain features of the photographic system needed to meet depth of field and resolution requirements necessary for the precise measurement of dendrite tip radii and growth velocities. A modified shadowgraphic technique will be utilized using a conventional flash lamp light source which provides the best compromise between the quality to the image and the exposure time. In addition, flat window glass growth chambers were designed to aid in the photographic testing. The photographic tests conducted by NASA Lewis at RPI on prototype rectangular glass chamber were quite successful in measuring tip radii and velocities at supercoolings up to 0.8 C. Subsequent tests will be conducted at NASA Lewis using the engineering model currently being

developed at RPI.

Data Analysis and Reduction

In conjunction with the photographic tests, a data analysis and reduction system has been developed concurrently in order to analyze the negatives from the various photographic systems. The system, consisting of a Ram Optical Instrument (ROI) optical measurement microscope (linear resolution - 1 micron), an oscilloscope and an IBM XT computer, has been assembled to evaluate objectively the quality of an image. The output of the microscope's camera is fed into the oscilloscope enabling the relative intensity of the image to be displayed. Three different types of measurements can be made to assess the quality of a particular dendritic image. Growth velocities are calculated by measuring the distance a dendrite has advanced between successive frames and the time between those frames and compared to ground based data. Secondly, an edge function width is measured with the aid of the oscilloscope. The edge function is defined as the distance over which the intensity of image falls from white (dendrite) to black (background) and may be equated to the uncertainty of the actual position of the edge. Data points around the edge of the dendritic image are measured and fit to a parabola using standard multiple regression routines. Tip radius may then be calculated and compared to the ground based data. The ROI optical microscope has been interfaced to an IBM XT and communication software has been developed allowing the direct input of data from the microscope into the computer. In addition, digital image processing of the images is currently under development to assist further in the objective evaluation of the photographs.

The data analysis system has been used to determine tip radius and growth velocity using the negative films obtained from the photographic tests conducted by NASA Lewis at RPI. The results obtained from these tests are in good agreement with the previous ground based data and indicate that our present data analysis system in combination with the NASA photographic system is effective in providing us with the required data for the flight experiment.

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Orbital Processing of Aligned Magnetic Composites

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November 1987 - October 1989

Objectives: The objectives of this program are to: (1) identify and quantitatively evaluate the influences of gravitationally driven thermal-solutal convection on contained plane front solidification of binary eutectic, off-eutectic, and peritectic magnetic composites; (2) evaluate the effectiveness of micro-g processing as a means of damping thermosolutal convectional; and (3) evaluate the uniqueness of micro-g processing relative to the best means of terrestrial convection damping.

Research Task Description: Three flight experiments have been conducted in which aligned, two-phase, magnetic composites were grown in Automated Directional Solidification Furnace (ADSF) Systems. The ADSF systems use the Bridgman-Stockbarger plane front directional solidification technique. This consists of translating a thermal gradient, which includes the solidification temperature, at a constant velocity down the length of a stationary sample (directional) under thermal conditions such that the solidification interface is flat (plane front solidification).

The first and last of these experiments was conducted in the Low Temperature Automated Directional Solidification Furnace System (ADSF-1), which was integrated into the mid-deck of Space Shuttle "Discovery" on Missions 51-G and STS-26, respectively. The second of these experiments flew in the High Temperature Automated Directional Solidification Furnace System (ADSF-2) on the Material Science Laboratory Carrier (MSL-2) in the payload bay of the Space Shuttle "Columbia" on Mission 61-C. The 51-G experiments studied off-eutectic Bi-Mn directional solidification and Soret diffusion, the STS-26 experiments studied eutectic Bi-Mn directional solidification and Soret diffusion, whereas the 61-C experiments studied Co-Sm eutectic solidification and thermal undercooling.

The relationships between the gravity vector, heat transfer, level of thermo-solutal convection and solidification processing parameters, are being studied terrestrially by varying the orientation of the gravity vector during solidification processing and by employing in-situ thermal measurements and interface demarcation techniques. These experimental results are compared with existent models of: heat flow, eutectic solidification, and off-eutectic solidification. In addition, a thermal model for the Bridgman-Stockbarger solidification technique including sample, ampoule, and translation, has been developed, and solidification models for eutectic, off-eutectic, and peritectic solidification with partial mixing in the melt, have been derived. The level of natural thermo-solutal convection is also varied by applying magnetic field (transverse and longitudinal) damping (MFD). Studies varying the thermosolutal driving force for convection and comparative analyses with magnetically damped and microgravity damped samples identify the role of gravitationally driven convection in Bridgman-Stockbarger plane front solidification.

Relationships between solidification processing parameters (including gravity vector), microstructure, macrostructure, chemistry as a function of fraction solidified, crystal structures, and magnetic properties have also been developed. Microstructure and macrostructure are analyzed using quantitative metallographic techniques. Chemistries

are determined using chemical spectrophotometric absorbance, x-ray fluorescence, magnetic, and microprobe analyses. Crystallography is studied using x-ray diffraction and Laue back reflection techniques. Magnetic measurements, which are sensitive to all of the above experiment and structural parameters, have been used as a sensitive means to determine the impact of gravitationally driven convection and convective heat transfer on an important physical property.

Micro-g results from Mission 51-G have shown that diffusion controlled growth ($k_{eff} = 1$) can be achieved orbitally, whereas it appears unachievable terrestrially, even using MFD. The diffusion-controlled results in micro-g, unexpectedly showed a greatly enhanced contribution of Soret diffusion to the chemical macrosegregation. This result was shrouded terrestrially by gravitationally driven convection.

Results from Mission 61-C experiment indicated that the damping of the thermosolutal convection was comparable to that of the 51-G experiment, diffusion-controlled growth having been achieved. Surprisingly, in a portion of the sample which free-cooled, the morphology noted can only be explained on the basis of significant thermal undercooling of the melt. Attempts to reproduce this morphology terrestrially have only succeeded under rapid solidification conditions.

The latest flight experiment directionally solidified a Bi-Mn eutectic (0.72 w/o Mn) at 1.0 cm/h, with an imposed thermal gradient of approximately 100K/cm. The eutectic rod and inter-rod dimensions will be measured and the interface undercooling estimated, in order to determine whether previously noted microstructural refinement during micro-g processing results from increased interface undercooling, a decreased transport coefficient, or both. In addition, Bi-Mn off-eutectic (0.60 and 0.45 w/o Mn) samples, which were held for 8 hours in a thermal gradient of 100 C/cm and then radially cooled, will be chemically analyzed to further refine the value of the Soret Diffusion coefficient.

The one-g experimental studies, in conjunction with theoretical analyses and experimentally determined thermophysical property measurements, will serve as a comparative base from which to evaluate the effectiveness of micro-g processing as a means of achieving diffusion-controlled growth of eutectic, off-eutectic, and peritectic composite crystals. Comparative analyses between the micro-g processed and the one-g damped results will determine the uniqueness of the orbital processing.

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Larson, D. J., "Materials Processing in Space," Materials Engineering Department Seminar, RPI, October 12, 1988.

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Larson, D. J., "Anomalous Thermal Undercooling in the Microgravity Environment," World Materials Congress, ASM/TMS Annual Meeting, Chicago, IL, September 26, 1988.

Solidification Fundamentals

Case Western Reserve University
Dr. V. Laxmanan
Professor John F. Wallace
NAG3-417 (NASA Contact: Tom Glasgow, LeRC)
March 1983 - November 1988

Objectives: The objective of this research, initiated in March 1983, was to obtain a fundamental understanding of gravitational during solidification of metals and alloys. Experimental work carried our in this program can be divided into three major categories. First, experiments in support of a Space Shuttle experiment on macrosegregation behavior in Pb-Sn alloys. Second, experiments aimed at obtaining a somewhat more fundamental understanding of dendritic and cellular growth, using a directional solidification apparatus. Third, experiments aimed at understanding the influence of undercooling on macro and micro segregation behavior in bulk samples (> 20 grams) of binary Pb-Sn alloys.

Research Task Description: The Space Shuttle experiment which was originally scheduled for the D-1 mission in 1985 and has since been cancelled since NASA Headquarters has decided to discontinue use of the General Purpose (Rocket) Furnace (GPF). Details of this work have been summarized in previous reports. The results obtained in our bulk undercooling experiments (sample size > 20 grams) with Pb-Sn alloys, in collaboration of Mr. Henry deGroh, have been summarized elsewhere. This report is aimed at summarizing the recent results using the directional solidification apparatus.

The central aim of the directional solidification experiments were to obtain simultaneous measurements of tip radii, primary dendrite arm spacings and tip temperature measurements during growth of a cellular or dendritic array in an externally imposed positive temperature gradient. Experiments were carried out with binary Sn-Pb alloys with various Pb contents: 5 wt%, 10 wt%, 15 wt%, 20 wt% and 25 wt%. All experiments were carried out with a nearly constant thermal gradient (of 160 to 170 K/cm) and various constant growth rates, varying from 1.0 micron/sec to about 60 microns/sec. Solidification was vertically upwards, against gravity. Thermal gradients and the dendrite tip growth rates were measured by recording the temperature trace of two thermocouples embedded with the melt and located a fixed distance apart.

It was found that the dendrite tip growth rate, R, determined from the trace of the two thermocouples, is usually higher than the withdrawal speed, V. The difference between V and R increases with increasing withdrawal speed. The measured dendrite tip temperatures, tip radii and primary spacings were used to test the predictions of various dendrite growth models. According to the model proposed by Laxmanan, a plot of the dendrite tip undercooling versus tip radius (or primary spacing) reveals a rather shallow minimum in the tip undercooling. Hence, the tip undercooling remains very close to the theoretical minimum value for a broad range of tip radii (or primary spacing). It is found that the experimentally measured tip radii and spacings fall within this range where the tip undercooling exhibits a "broad physical minimum". However, the measured tip radius and spacing are found to be much higher than the "mathematical minimum value". In most cases the measured tip radii and spacings are also much higher than the values obtained from marginal stability considerations. A re-examination of experimental data in other alloy systems (notably SCN-Ace, Al-Cu, Pb-Au alloys) also reveals similar results. In these alloys, tip radii an primary spacings seem to fall in the

sides of the mathematical minimum, but always within the broad range of a physical minimum in the tip undercooling. Several publications detailing the findings are now in preparation.

Publications

DeGroh, H. C. and Laxmanan, V., "Macrosegregation in Undercooled Pb-Sn Eutectic Alloys" in *Proceedings on Conference on Solidification Processing of Eutectic Alloys*, (D.M. Stefanescu, G.J. Abbaschian, and R.J. Bayuzick, eds.), TMS, 1988, pp. 229-242.

DeGroh, H. C., and Laxmanan, V., "Bulk Nucleation and Macrosegregation of Pb-Sn Alloys," Met. Trans., 1988 (submitted).

Wang, L., Laxmanan, V., and Wallace, J. F., "Gravitational Macrosegregation in Small Unidirectionally Solidified Pb-Sn Alloy Ingots," Met. Trans., 1988 (submitted).

Laxmanan, V., "Morphological Transitions during Rapid Solidification Processing: A Re-examination of Fundamental Validity of the Absolute Stability Concept of Mullins and Sekerka," Acta Met., 1988 (accepted).

Casting and Solidification Technology (CAST)

University of Tennessee Space Institute
Dr. Mary Helen McCay
Dr. T. Dwayne McCay
Dr. Robert Owen
Ronald Porter
Montgomery Smith
NAS8-37292 (NASA Contract: R.C. Ruff, MSFC)
December 1986 - December 1990

Objectives: The objectives of research are: (1) to determine the influence of gravity on the fluid flow and nucleation that occurs during casting, and (2) to investigate the solidification and coarsening processes of dendrite arms and their subsequent influence on the grain structure in castings.

Research Task Description: The purpose of the investigation is to study the directional solidification of metal-model materials under low-gravity conditions. In particular, the inverted density layer and the thermal and solutal fields ahead of growing interface will be analyzed. This investigation is an extension of previous low-g studies done on Space Processing Applications Rocket (SPAR) and KC-135 flights. To complete these studies, longer periods of low-g are required in order to allow solidification to occur at slower and more controllable rates. Therefore, detailed analysis will be made of fluid motion near the solidification interfaces using the optical techniques (Schlieren, shadowgraph, interferometry and holography) available in the ground based laboratory and in the Fluid Experiment System on IML-2. To aid in separating the thermal and solutal profiles, thermocouples will be placed at intermittent locations along the cuvette wall. The temperature measurements will enable the investigators to mathematically separate the thermal and solutal effects on the interferograms.

Progress to Date: A double optics system was assembled that provides confocal processing and particle tracking capabilities. The onset of convective instabilities within th inverted layer are being documented with this apparatus. A cuvette design that would accommodate shrinkage and expansion of the fluid wwas completed. An instrumented cuvette was assembled and operated under typical solidification conditions. Comparisons of concentration and temperature fields were made with optical and analytical predictions. A KC-135 flight rig was assembled incorporating a confocal optical system and VCR data recording. The advanced fluid thermal model has progressed continually and a graphics capability has been added so the solidification process can be viewed in real time as it is being calculated.

Publications:

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McCay, T. D., McCay, M. H., Lowry, S. A., and Smith, L. M., "Convective Instabilities during Directional Solidification," J. Thermophys. & Heat Transf., 1988 (accepted).

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Presentations:

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3. FLUID DYNAMICS AND TRANSPORT PHENOMENA

Dynamic Thermophysical Measurements in Space

National Institute of Standards and Technology
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W-16,247 (NASA Contact: R. Crouch, NASA HQ)
January 1, 1988 - October 31, 1988

Objectives: The objective of this research is to develop techniques for the millisecond-resolution dynamic measurement of selected thermophysical properties (heat of fusion, heat capacity, surface tension, electrical resistivity, hemispherical total and normal spectral emittances) of high-melting-point electrically-conducting solids and liquids at temperatures above 2000°K in a microgravity environment. The initial goal is the development of a technique for electrically-conducting materials, which when completed, will enable, for the first time, the extension of accurate thermophysical measurements to temperatures above the limit (melting point) of the highly-successful ground-based millisecond-resolution experiments. The longer-range goal is the development of a technique for electrically nonconducting materials (ceramics), which when completed will enable the accurate measurement of the thermal properties of these important materials at high temperatures.

Research Task Description: The technique for electrically-conducting materials is based on rapid resistive self-heating of the specimen up to its melting point and above in about one second, and performing measurements of the experimental quantities with millisecond resolution. The initial phase of this research requires establishment of the geometrical stability of a specimen when heated rapidly to temperatures above its melting point in a microgravity environment. A test equipment package has been designed and constructed which permits rapid heating of specimens in various geometrical configurations (solid cylnsrical, tubular, triaxial, etc.). This system has been flown several times on board the KC-135 aircraft. Parallel with the experimental work, theoretical work is underway in this direction to understand the behavior (geometrical stability) of the liquid specimen, and as a result, optimize the specimen geometryt and the operating conditions of the overall system. The next phase of the work is to add new capabilities to the system for the rapid and accurate measurement of electrical power, temperature, and temperature gradients in the specimen, taking into account the requirements for operation in a microgravity environment for extended periods of time. Significant progress has been made in this direction, including construction and partial testing of two high-speed pyrometers (multiwavelength and spatial scanning). The system will be used to demonstrate the applicability of the technique to performing definitive measurements of selected thermophysical properties of one or more refractory metals, such as niobium, tungsten, etc., at and above its melting point in a microgravity environment.

The technique for electrically nonconducting materials is based on performing measurements (with millisecond resolution) of the experimental quantities during the free cooling of a specimen from an initial high temperature state. The initial high temperature state may be achieved by heating the specimen either with a laser or with concentrated solar energy. The time-resolved measurements of the temperature and power radiated from the specimen are required for this technique. Multiwavelength and sptial scanning pyrometers for measurements, in conjunction with a pyroelectronic detector for power measurements, are to be utilized. This technique has the potential of yielding heat capacity, hemispherical total emittance, and thermal diffusivity of high-temperature liquid materials from data obtained in microgravity experiments.

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Progress to Date: Analytical wotk has shown that a triaxial configuration for the tubular specimen and the current return path can provide a better geometrical stability for the molten specimen. Experiment chambers have been constructed, based on the triaxial design criteria, and microgravity-simulation experiments were performed on tantalum and copper specimens on board the KC-135 aircraft. The results indicated improvements in operation and showed that the macroscopic dimensional behavior of the specimen melt zone could be predicted by the analytical model, thus suggesting a new method for measuring surface tension. Surface tension of copper at its melting point was determined by this technique, which is in good agreement with the data reported in the literature.

Testing of the high-speed multiwavelength pyrometer was continued. Construction of the high-speed spatial scanning pyrometer was completed and its testing was begun. This pyrometer, the only one of its kind, can measure spectral radiance temperature at about 1000 points along a straight line (25 mm long) on the specimen; a complete cycle of measurements can be performed in about 1 ms. This pyrometer will permit measurements of temperature gradients in a rapidly heating and/or cooling specimen, providing data fo diagnostic purposes and for determination of thermal diffusivity, which will be a novel approach suitable for measurements at very high temperatures.

Heat of fusion of molybdenum was measured on the ground-based microsecond-resolution, pulse-heating facility to generate data for comparison in future experiments in a microgravity environment with the millisecond-resolution system.

Operational characteristics (time response, linearity, stability, reproducibility, etc.) of a pyroelectric detector were investigated utilizing a bench set-up. In addition, the detector was used to measure power radiated from a rapidly heating and cooling specimen in the ground-based millisecond-resolution system. The preliminary results demonstrated the feasibility of using pyroelectric detectors for the measurement of radiated power from a specimen under transient conditions.

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Cezairliyan, A., and Miller, A. P., "A Dynamic Technique for Thermophysical Measurements at High Temperatures in a Microgravity Environment," Int. J. Thermophys., 1988 (in press).

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Surface Tension Driven Convection

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Professor Simon Ostrach
Professor Y. Kamotani
NAG3-570
(NASA Contact: T.P. Jacobson, LeRC)
January 1988 - October 31, 1988

Objectives: The objective of the investigation is to design a thermocapillary experiment to study the transient and steady-state flows in the long-duration low-g environment of the Shuttle.

Research Task Description: The experiment consists of a circular container (5 cm dia. and 5 cm deep) filled with silicone oil, heating systems, and a data acquisition system. The fluid free surface will be heated locally by a CO_2 laser or by a submerged circular heater placed at the center. The resultant temperature variation along the free surface will generate thermocapillary flow in the container. The flow field will be studied by a flow visualization technique and the temperature distribution along the free surface, which is important because it determines the driving force of the flow, will be measured by a thermography technique. The surface heat flux distribution, the heating level, and the static free surface shape will be varied to study their effects on the nature and extent of the flows. Two series of experiments are planned. In the first one, the basic thermocapillary flow will be studied and attempts will be made to obtain oscillatory thermocapillary flow. In the second series the oscillation phenomenon will be studied in detail because it is considered to be an important aspect of thermocapillary flow. Ground-based and drop tower experiments together with a numerical analysis have been made to provide base data and to ensure that the operating condition and the configuration will lead to flows that can be reasonably observed and measured.

Progress to Date: The engineering design of the test cell and various support systems and the development of the diagnostic techniques are being done at the NASA Lewis Research Center. Ground-based studies are continuing at CWRU.

Publications

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The Mathematical and Physical Modelling of Electromagnetically Drive Fluid Flow and Associated Transport Phenomena in Contained and in Containerless Melts

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Dr. T. Kang
NAG3-594
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Objectives: The objective of this research is to develop an improved fundamental understanding of electromagnetic, heat flow and fluid flow phenomena in levitation melted specimens under both earthbound and microgravity conditions. The main motivation of this work is twofold: (1) a number of fundamental hydrodynamic and electromagnetic issues may be uniquely addressed in this manner, and (2) levitation melting is a key ingredient of many materials processing experiments in space, thus the present project provides an important support function for this effort.

Research Task Description: The current research pursues three complementary directions (1) extensive computational work is being carried out to predict the electromagnetic force field, the velocity field and the temperature fields in electromagnetically stirred (positioned) metallic specimens. An important novel feature of this effort is that an allowance is being made for the behavior of free surfaces and free surfaces deformation; (2) experimental work has been carried out to measure and predict electromagnetically driven flows in a molten Woods metal pool due to the passage of current between two electrodes; and (3) calculations are being carried out to support a planned in-flight experiment aimed at measuring the viscosity of undercooled melts using a levitation melted specimen.

Progress to Date: Important milestones of the research include the following: (1) the development of a general methodology for computing electromagnetic force fields and the corresponding melt velocity fields in complex geometries; (2) the experimental measurement of electromagnetically driven flows in Woods Metal Melts, for both steady and fluctuating electromagnetic force fields; and (3) the development of a new initiative for measuring the viscosity of undercooled melts using levitation melted metallic droplets.

Publications

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Production of Large-Particle-Size Monodisperse Latexes in Microgravity

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Dr. F. J. Micale
Dr. M. S. El-Aasser
Dale Kornfeld, MSFC
NAS8-32951
(NASA Contact: V. Yost, MSFC)
January 1988 - January 1989

Objectives: The objectives of this research are: (1) to produce large-particle-size monodisperse polystyrene latexes in microgravity in sizes larger and more uniform than can be manufactured on Earth; and (2) to develop a model for a heterogeneous chemical reaction in microgravity.

Research Task Description: The uniformity of the particles made in microgravity was better than that of the most uniform particles made on earth. The coefficients of variation of the 5-30 micrometer space particles were 1.0-1.4%; those of the best 10-100 micrometer particles made on earth were 2.0-2.5%, with others ranging up to 5%.

The 5-30 micrometer latexes made in microgravity formed negligible maounts of coagulum; the corresponding ground-based control latexes formed increasing amounts with increasing particle size up to 18 micrometers.

The 10 micrometer two 30 micrometer space latexes were accepted by the National Bureau of Standards as Standard Reference Materials, to make them the first products made in space for sale on earth. Almost half of the 10 micrometer samples have been sold; the 30 micrometer particles have only recently been offered for sale.

The National Bureau of Standards has found that the 5, 10, and 30 micrometer space particles are more perfect spheres than the corresponding particles made on earth.

Since the last flight experiments, polymerization recipes have been developed which give particles as large as 100 micrometers with tolerable levels of coagulum and coefficients of variation of 2.5%. The successive seeded polymerization grew 0.13 micrometer seed particles to 100 micrometers in 18 steps.

The rates of polymerization in microgravity were the same as on earth within experimental error. The kinetics of polymerization kinetics were those of microsuspension polymerization, with each particle polymerizing as a tiny bulk reactor. Thus, this system provides an excellent model for a heterogeneous chemical reaction in space.

During the successive seeded polymerization to 100 micrometers, it was discoverd that certain polymerizations gave uniform nonspherical particles instead of spheres. These nonspherical particles formed by the contraction upon heating of the monomer-swollen crosslinked spherical network upon heating and the solidification of new domains by polymerization.

Systematic methods were developed to produce uniform non-spherical particles (e.g., ellipsoidal and egg-like singlets, asymmetric and symmetric doublets, and ice cream cone-like and popcorn-like multiplets). Systematic investigation of these polymerizations

showed that the degree of phase separation increased with increasing degree of crosslinking of the seed particles, monomer/polymer swelling ratio, polymerization temperature, and seed particle size; it decreased with increasing divinylbenzene concentration in the swelling monomer.

A thermodynamic analysis of the swelling and polymerization of latex particles showed that the network contracted upon heating because of the balance between the elastic-retractile force of the crosslinked network, the polymer/water interfacial tenion force that restricts the swelling of the particles, and the monomer-polymer mixing force that occurs upon swelling of the particles with monomer. This balance of firces gave calculated latex particle swelling ratios that were in good agreement with the experimental values.

Publications

Sheu, H. R., El-Aasser, M. S., and Vanderhoff, J. W., "Uniform Nonspherical Latex Particles as Model Interpenetrating Polymer Networks," Polym. Mat. Sci Engr. 59, 1185 (1988).

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4. BIOTECHNOLOGY

Cell Partition in Two Polymer Aqueous Phases

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Dr. J. Milton Harris, UAH

When aqueous solutions of two different polymers are mixed above certain concentration they frequently form immiscible, liquid, two-phase solutions. Each of these phases usually consists of more than 90 percent water and can be buffered and made isotonic by the addition of low molecular weight species. If a cell or particle suspension is added to such a system in 1-g, then shaken, the system demixes rapidly and cells are usually found to have partitioned unequally between one of the phases and the interface. This preferential partition behavior can be used as the basis of a separation procedure for differing cell populations since partition in these systems is determined directly by cell membrane properties. Such systems are being employed in many countries to carry out biotechnical separations and continuous bioconversion extractions.

By manipulating the composition of the phase systems, separation on the basis of a variety of molecular and surface properties have been achieved, including membrane hydrophobic properties, cell surface charge and membrane antigenicity. When the results of these separations are compared with predictions based on thermodynamic measurements made on single cells in the systems, it is found the separation efficiency is orders of magnitude lower than the thermodynamic limit. This may be due in part to cell sedimentation but other factors are undoubtably also responsible for this discrepancy. Displacement of cells from their location of lowest equilibrium free energy may be due to the chaotic hydrodynamic environment in which the cells are imbedded during convection-driven phase demixing. To test this idea we are aiming at performing cell separations in microgravity where demixing occurs in the absence of convection, creating a more quiescent hydrodynamic environment. In order to carry out such experiments information regarding the determinants of demixing rates and the disposition of demixed phases in the absence of buoyancy effects is required. Studies conducted onboard KC-135 aircraft during parabolic manueuvers and by Senator E.J. Garn onboard Shuttle flight STS-51D have indicated that in low-g aqueous polymer two-phase emulsions demix by a slow coalescence process. Very low fluid shear is present, suggesting that low-g partition may be able to resolve cell subpopulations unobtainable, by any method, on Earth.

In low-g, phase emulsions demix to yield one phase floating like an egg yolk, surrounded by the phase which preferentially wets the container wall. Current research is aimed at controlling the rate of demixing and final disposition of the phases via both passive means (e.g., altered chamber geometry or polymeric wall coatings with different wetting properties) and active means (electrophoresis of the phase whose interfaces exhibit zeta potentials). In addition, variables such as interfacial tension, phase volume ratios and phase viscosity, are being studied to better understand their influence on demixing of the phases on both low-g and 1-g. Many of these variables were successfully investigated in another passive demixing Phase Partitioning Experiment (PPE) flown on STS-26. The demixing processes under study are relevant to a variety of demixing phenomena in materials processing.

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Protein Crystal Growth in a Microgravity Environment

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NAS8-36611 (NASA Contact: John Price, MSFC)
January 1987 - January 1988

Objectives: The long range objective for this research task is to develop systematic and reliable techniques and hardware for growing protein crystals in space. Studies will be performed to evaluate the potential for enhanced protein crystal growth under microgravity conditions. Fundamental studies of protein crystal growth, both on the ground and in space will be performed in order to identify the major parameters that affect protein crystal growth. The fundamental studies will be used to define the science requirements to be incorporated into advanced hardware that permit investigators to dynamically monitor and control the major parameters in protein crystal growth.

Research Task Description: This research program involves a multidisciplinary effort to produce protein crystals in space of sufficient quality and size to permit molecular structural characterization by X-ray crystallography, while simultaneously providing basic ground-based experimental and theoretical supporting research to develop a better understanding of protein crystal growth and to determine if gravity plays a limiting role in the growth process. Beginning with the Apollo program and extending into the Spacelab program, it has been demonstrated that the microgravity environment can provide stable growth conditions than can result in crystals with improved homogeneity and fewer defects. In this program, a variety of proteins will be crystallized on space shuttle flights over a three-year period. Optimum techniques for reliably growing protein crystals under microgravity conditions will be developed. Initially, emphasis will be placed on modified vapor diffusion techniques such as the "hanging drop method" that is widely used for ground-based studies in protein crystal growth. Long-range plans include development of new methods for growing protein crystals, based upon the experimental and theoretical studies performed as part of this research program. A major goal is to develop advanced hardware that will be available for use on the Space Station.

Progress to Date: Protein crystal growth hardware for use on the space shuttle has been developed during the past two years, and this hardware will be flown in the middeck on Shuttle flight STS-26. The hardware was constructed at Teledyne Brown Engineering Company, based upon the experience gained with four middeck flights in 1985 and 1986. Prototypes of this flight hardware have been extremely tested during the past year in laboratory studies at UAB and by co-investigators at various academic, government and industrial laboratories in the U.S. The hardware that has been developed for STS-26 will replace a middeck locker, and will permit 60 protein crystal growth experiments to be performeed on that shuttle mission.

Along with the hardware development program, a number of basic studies in protein crystal growth have been completed. Particular emphasis has been placed on methods for detecting initial nucleation events, using laser light scattering and other optical techniques. As part of this project, investigators at the University of Alabama in Huntsville have determined the first phase diagram for crystallization of a protein, using the widely studied lysozyme system. Other work supported under this contract at Georgia Tech has led to the development of a nautomated system for dynamic control of vapor diffusion equilibration rates. These studies will be combined together to define

hardware and techniques for dynamic control and monitoring of protein crystal growth parameters.

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Purification of Bioactive Pituitary Growth Hormone Cells and Pituitary Growth Hormone Molecules

Pennsylvania State University
Dr. Wesley C. Hymer
Dr. Richard Grindeland, ARC
Dr. Wayne Lanham, MDAC
Dr. Dennis Morrison, JSC
NAS9-17416
(NASA Contact: D.R. Morrison, JSC)
December 1, 1984 - December 1, 1988

Objectives: The objectives of the research are: (1) development, validation and establishment of sensitive bioassays for rate and human GH; (2) isolation of GH cell subpopulations and subcellular GH containing particles; and (3) isolation of GH variants from the mammalian pituitary which have high biological activities; and (4) isolation and characterization of GH cell subpopulations.

Research Task Description: To meet the first goal, we are developing two in vitro GH bioassays. One uses a macrophage cell line that, when exposed to increasing concentrations of hormone, responds by releasing oxygen metabolites (superoxide) that can be quantified. The other uses rat epiphyseal chondrocytes, and quantifies chondrioton sulfate production in response to the hormone. To meet the second goal, SDS-PAGE and HPLC are being used to isolate GH variants. To meet the third goal, centrifugal and electrophoretic techniques are being used to idnetify and characterize subpopulations of pituitary secretory granules. To meet the fourth goal, centrifugal and laser flow cytometric techniques are being used to isolate subpopulations of GH producing pituitary cells.

Progress to Date: Release of superoxides from macrophages into the culture medium is linearily related to the amount of GH added (10-100 nanograms). The response is dependent upon serum concentration, kinetics after GH exposure and PMA levels,. Additional validation is in progress. Preliminary results with rat chondricytes show increased responses to added GH, but these are not linear.

Considerable progress has been made on the isolation and characterization of high molecular weight GH forms. We know that these forms are composed of S-S linked monomers of 20 KD and 22 KD. Aggregation is not random. Reduction with mercapto-ethanol enhances GH immunodetection. These forms are contained in secretory granules and are released into the medium of cultured cells. We have found that the increased immunoassayability on chemical reduction which offers strong support for the idea that the high molecular weight forms represent a bioactive form of GH. Finally, we are characterizing GH cells prepared from single rat pituitary glands in terms of (a) their position in the gland, (b) their laser flow light scatter character, and (c) their GH release capacities. Results to date show heterogeneity in responses.

Publications

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Todd, P., Hymer, W. C., Morrison, D. R., Goolsby, C. L., Hatfield, J. M., Kunze, M. E., and Motter, K., "Cell Bioprocessing in Space: Applications of Analytical Cytology," in *Proceedings of Ninth Annual Meeting IUPS*, Commission on Gravitational Physiology Czechoslovakia, September 28 - October 1987. (in press)

Kidney Cell Electrophoresis in Microgravity

National Institute for Standards & Technology
Dr. Paul Todd
NAS9-17431
(NASA Contact: D.R. Morrison, JSC)
May 1, 1985 - July 31, 1989

Objectives: The objectives of this research task are: (1) provide ground-based cell electrophoresis technology and cell culture support for electrophoretic purifications of cultured human embryonic kidney cells in microgravity; (2) develop flow cytometric methods for analyzing and sorting of human embryonic kidney cells; and (3) perform biophysical analyses of purified cultured human embryonic kidney cells returned from space flight.

Research Task Description: To accomplish the first objective, cultured human embryonic kidney cells, in early passage will be subjected to continuous flow electrophoresis. The effects of content of divalent cations, antibiotics, and neutral additives will be studied in low conductivity electrophoretic separation experiments. Cells from various lots will be examined and compared with respect to their ability to multiply, to produce plasminogen activators, to differentiate morphologically, and to retain differentiated function in vitro.

In response to this objective it was found that: (1) kidney cells migrate in continuous flow electrophoresis according to their electrophoretic mobility as determined by analytical electrophoresis (laser grating anemometry) before and after separation, (2) calcium ions at moderately high (20 mM) concentrations can neutralize the negative charge in kidney cells, (3) Ficoll, which is neutral, increases electrophoretic mobility, and (4) one cell lot, designated HEK-1593, has a high plating efficiency, high growth rate, high plasminogen activator production, and relatively narrow electrophoretic mobility distribution.

To accomplish objective two, a laser flow cytometer will be used to quantify cells that produce plasminogen activator on a one-by-one basis by staining them with fluorogenic amide substrates that release 4-methoxy-2-naphthylamine which can be precipitated intracellularly by the addition of 5-nitrosalicylaldehyde. The percent plasminogen activator producing cells will be quantified in early-passage cultures and purified cell suspensions by this method. The light scattering signature in flow cytometry of electrophoretically purified cell subpopulations will be determined in order to establish whether or not there exist basic morphological criteria that may also be used as a means of cell purification by viable cell sorting. A flow cytometer will also be used to examine the relationship between cell surface charge and cell function by staining simultaneously with fluorescent poly-L-lysine and plasminogen activator fluorogenic substrates.

In response to this objective, it was found that: (1) about 50% of a population of kidney cells fluoresced brightly in the EPICS V cytometer when stained for intracellular plasminogen activator, (2) about 50% of this fluorescence intensity was lost after cells had spent 3 days in "production medium," which stimulated plasminogen activator secretion, (3) cells from different electrophoretic sub populations have different light-scattering signatures as determined by flow cytometry, and (4) in model cell types, polysine staining of the cell surface is a dynamic process, but staining can be shown to be proportional to sialic acid on the cell surface when cells are fixed.

To accomplish objective three, post-flight biophysical and biochemical analyses of cells purified in space, the above mentioned methods will be used. The percent cells producing each of the three different types of plasminogen activator will be identified in each fraction using specific markers in flow cytometry. Cell life cycle analysis using DNA staining and flow cytometry of cells from each purified fraction will be used to reveal which fractions retain reproductive potential. The electrophoretic mobility of cells from each purified fraction will be confirmed on the basis of analytical cell electrophoresis using an automated electrokinetic analyzer.

In response to this objective, cell life cycle analysis was performed on the basis of propidium iodide staining of nuclear DNA followed by flow cytometry with no unusual findings, and three electrophoretic fractions were compared with respect to reproductive potential with no obvious differences observed among them. The electrophoretic mobilities of kidney cell populations separated by continuous flow electrophoresis on the ground were measured and found to be consistent with their migration distances. A fibrinagarose overlay method was developed for detecting plasminogen activators separated according to molecular weight by electrophoresis. The method successfully distinguishes among high- and low-molecular weight urokinase and tissue plasminogen activator (t-PA). No new space experiments could be performed during the project period, but analyses of frozen samples separated on space shuttle flight STS-8 continues.

Progress to Date: Emphasis during the last calendar year has been placed on the development of biophysical analytical tools for the pre-flight and post-flight analysis of kidney cell populations.

Publications

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5. GLASSES AND CERAMICS

Containerless Processing of Glass Forming Melts in Space: Critical Cooling Rates and Melt Homogenization

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Dr. Delbert E. Day
NAS8-34758
(NASA Contact: V. Fogle, MSFC)

Objectives: The opportunity of melting and solidifying materials without physical contact with containers and the absence of convection are two major advantages of the microgravity environment. The present research program is designed to utilize these advantages for processing multicomponent glass forming melts in space. The main objectives are to: 1) obtain quantitative evidence for the suppression of heterogeneous nucleation/crystallization in containerless melts in microgravity, 2) study melt homogenization in the absence of gravity-driven convection, 3) develop the procedures for preparing precursor samples suitable for flight experiments, 4) perform comparative property analysis of glasses melted on earth and in microgravity, 5) determine the feasibility of preparing glass shells in microgravity for use as laser fusion targets, and 6) assess the operational performance of the acoustic levitator/furnace for processing multicomponent, glass forming melts in microgravity.

Research Task Description: If heterogeneous nucleation/crystallization of a melt is suppressed by containerless melting, then its critical cooling rate (R_c) for glass formation in microgravity will be less than its R_c on earth. The practical consequence of a smaller R_c in microgravity is an extension of the compositional limits for glass formation and the possibility of obtaining new, useful glasses by melting in microgravity. For samples forming glass after containerless melting in microgravity, the ratio of R_c on earth to the cooling rate (R) used in microgravity will serve as a quantitative measure of the degree to which glass formation is enhanced, or heterogeneous nucleation is suppressed. Ternary calcia-gallia-silica compositions, having different Rc values will be heated, melted, and quenched in microgravity in an acoustic levitator furnace. Selected physical, optical, thermal, and mechanical properties will be measured for glasses made in microgravity for comparison with the same properties of glasses made on earth. Glass spheres containing an irregularly shaped air bubble will be remelted in microgravity in order to examine the feasibility of producing glass shells of thin uniform wall thickness. Melt homogenization in the convection-free environment of microgravity will be investigated by observing the level of chemical homogeneity achieved in melts made from hot-pressed samples containing known inhomogeneities. The suitability of using hot-pressed samples for flight experiments in microgravity will be determined.

Progress to Date: Ground-based work which includes 1) preparation, microstructural characterization, and property measurements (density, porosity) of hot-pressed precursor samples for future flight experiments, 2) property measurements, including high temperature viscosity, of the glasses prepared on earth for compositions to be used in flight experiments, and 3) evaluating the feasibility of using sol-gel technique to prepare samples for flight experiments, were conducted during this period. The glass forming characteristics for several calcia-gallia-silica melts (candidate compositions for future flight experiments), while levitated aerodynamically and melted by a CO_2 laser were studied using hot-pressed precursors. The primary purpose was to simulate more closely on earth containerless glass melting in space and to obtain ground-based data under nearly identical conditions as will be used for experiments in microgravity. The effect of nucleating agents, surrounding atmosphere, and preannealing temperature and time on

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the crystallization of lithium-silicate and bismuth-germanate glasses was also investigated using differential scanning calorimetry (DSC). Knowledge of the crystallization characteristics of these two glass systems is relevant to their potential use in flight experiments and to understanding the extent to which heterogeneous nucleation is reduced in containerless melts in microgravity.

Publications

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Fluoride Glasses: Crystallization and Bubbles in Low Gravity

Rensselaer Polytechnic Institute
Dr. Robert H. Doremus
Contract No. 955870
January 1, 1987 - June 30, 1987

Objectives: The objective of this research is to study the influences of surface composition and structure on properties of zirconium fluoride glasses, vaporization, crystallization and chemical reactions. The ultimate objective is to determine the influence of containerless melting in microgravity on the properties and formation of glasses, as compared to melting in a container in earth's gravity.

Research Task Description: Zirconium fluoride glasses of different compositions are being melted and their vaporization, surface reactions, and crystallization studied. Typical glass compositions are (in mole%) ZBL, $62ZrF_4$, $33BaF_2$, $5LaF_3$, ABLAN, $53ZrF_4$, 20BaF, $4LaF_3$, 4A F_3, 20NaF; BaYbZnTh, $23BaF_3$, $27YbF_3$, $27ZnF_2$, $23ThF_4$. The influence of different melting conditions and atmospheres and subsequent heating at about 300 C in different atmospheres on crystallization and surface composition is being studied. Experimental tools are X-ray diffraction, Rutherford back-scattering, ellipsometry, mass spectroscopy, and electron microscopy.

Progress to Date: Surfaces of different fluoride glasses which reacted with different gases have been analyzed with Rutherford backscattering and ellipsometry. Composition profiles of different constituents have been measured, and are consistent with reaction control by interdiffusion in the glass.

Publications

Mathew, J. and Doremus, R. H., "Vaporization of Zirconium Fluoride Glasses," J. Non-cryst. Solids 102, 165 (1988).

Physical Phenomena in Containerless Glass Processing

Clarkson University
Dr. R. Shankar Subramanian
Dr. Robert Cole
NAS8-32944 (NASA Contact: V. Fogle, MSFC)
December 1977 - December 1989

Objectives: The objective of this investigation is to develop an understanding of fluid motion and bubble and droplet motion and interactions when drops containing bubbles are subjected to stimuli such as surface tension gradients, rotation, expansion and contraction, and oscillation.

Research Task Description: At this time, all of the research is ground-based. Flight experiments in an acoustic levitator are planned for execution in the future. The research task involves conducting experimental studies of bubble/drop motion in a temperature gradient and in rotating liquid bodies. Supporting theoretical models of ground-based experiments as well as planned flight experiments are also to be developed.

Experiments have been completed on bubbles migrating normal to plane solid surfaces in a vertical temperature gradient. Bubbles moving downward in a temperature gradient near a surface were found to move more rapidly than isolated bubbles. This counter-intuitive behavior was explained satisfactorily based upon the behavior of the disturbance velocity fields for body force driven versus thermocapillary motion. The experimental results were in agreement with predictions from theory.

Theoretical models were developed for determining the impact of surfactants on the thermocapillary migration of a drop. Also, theoretical models are being developed for predicting the migration velocity of a bubble within a drop in a space laboratory when a non-axisymmetric temperature field is imposed on the drop surface.

Progress to Date: Experiments on the motion of bubbles contained in freely suspended rotating drops are in progress.

Publications

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Ruggles, J. S., Cook, R. G., Annamalai, P., Cole, R., "Bubble and Drop Trajectories in Rotating Flows," Exper. Thermal and Fluid Sci. 1, 293-301 (1988).

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Presentations

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Ruggles, J. S., Cook, R. G., and Cole, R., "Microgravity Bubble Migration in Rotating Flows," *AIChE Spring National Meeting*, New Orleans, Louisiana, March 1988.

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6. COMBUSTION SCIENCES

Scientific Support for an Orbiter Middeck Experiment on Solid Surface Combustion

University of Kentucky
Professor Robert A. Altenkirch
Dr. S. Bhattacharjee
NAS3-23901 (NASA Contact: S. Olson, LeRC)
December 19, 1984 - December 20, 1992

Objectives: The overall objectives of the experiment are to: (1) determine the mechanism of gas-phase flame spread over solid fuel surfaces in the absence of any buoyancy induced or externally imposed gas-phase flow; and (2) improve the fire safety aspects of space travel.

Research Task Description: The spread of flame in the gas over the surface of a solid combustible involves in an essential way the transfer of heat from the flame to the solid fuel immediately ahead of it. This heat transfer is affected by the character of the gas-phase flow, and so the phenomenon of flame spreading under reduced gravity, in which the flow is generated by gasification of the solid combustible, is apt to be different from what occurs under the Earth's normal gravitational acceleration where the flow is largely buoyancy driven.

An experiment has been designed for the Middeck of the Space Shuttle to aid in understanding the process of flame spreading in the absence of a buoyancy-driven flow. A chamber approximately 0.035 m^3 in volume is to contain either a thin sample of a cellulosic material or a thick sample of polymethylmethacrylate and an oxidizing environment of O_2 and N_2. Samples will be ignited at one end, and the ensuing flame spread process will be filmed. The spread rate can be determined from the films, and surface and gas-phase temperatures just above the surface will also be recorded. A matrix of eight experiments to be carried out on the Middeck has been identified. These data will help to clarify the mechanism of forward heat transfer in the low-gravity flames.

The experimental apparatus has been constructed at NASA's Lewis Research Center and tested in the Drop Tower facilities. Testing was carried out at the University of Kentucky while the Principal Investigator served on the faculty of that institution. Methods of data reduction are being developed as are theoretical analyses of reduced-gravity flame spread problem. Results to date show that measured spread rates over tin cellulosic fuels are less at microgravity than for downward spread in normal gravity. Theoretically predicted microgravity spread rates not accounting for radiation heat loss from the flames are for infinitely-fast, gas-phase chemistry generally a factor of approximately five times what is measured For finite-rate, gas-phase chemistry, the predicted values are approximately three times those measured.

Progress to Date: Computational methods for determining gas-to-solid heat flux from experimental solid-surface temperature data for thin solid combustibles have been developed and applied to experimental drop tower results. Spread rates with an without consideration of solid-surface reradiation for finite-rate chemistry can now be computed from the theoretical modelling effort. Comparison of computed spread rates with those obtained from the short-duration, drop tower experiments showed computed spread rates neglecting surface radiation to be higher than meadured spread rates. Surface radiation reduces computed spread rates such that they are closer to those measured experimentally. Computed and experimental spread rates generally follow the same trends as parameters vary except for pressure. Computed spread rates neglecting surface

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readiation tend to decrease slightly with an increase in presure while experimental ones show the opposite trend. The inclusion of radiation may remove this discrepancy.

Publications

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Presentations

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Particle Cloud Combustion Experiment

University of California, San Diego
Dr. A. L. Berlad
Dr. V. Tangirala
NAS3-24639 (NASA Contact: Howard Ross, LeRC)
September 1, 1985 - August 31, 1988

Objectives: The principal objectives of this microgravity experimental program are to obtain flame propagation rate and flame extinction limit data for several important premixed, quiescent particle cloud combustion systems under near zero-gravity conditions. The data resulting from these experiments are needed for utilization with currently available flame propagation and extinction theory. These data are also expected to provide new standards for the evaluation of fire hazards in particle suspensions in both Earth-based and space-based applications. Both terrestrial and space-based fire safety criteria requires the identification of the critical concentrations of particulate fuels and inerts at the flame extinction conditions.

Research Task Description: The Particle Cloud Combustion Experiment (PCCE) employs long flame tubes within each of which a uniform quiescent cloud of particlesis to be suspended in reduced gravity. Particulates under study include the fuels lycopodium, cellulose and coal, as well as a number of inert particulates. Ground-based supportive studies include the use of the LeRC drop tower and Learjet research facilities as well as the laboratories at the University of California, San Diego. Laboratory experiments include studies of particlee cloud mixing, particle-particle interactions, pyrolysis-vaporization kinetics, and the radiative attenuation properties of individual particles and of particle clouds LeRC drop tower and Learjet facilities are employed to study particle cloud mixing and particle cloud combustion phenomena in reduced gravity.

Progress to Date: LeRC drop tower and Learjet facilities have been used successfully to study low-gravity particle mixing and flame propagation phenomena supported by quiescent clouds of lycopodium particulates. One mode of quasisteady flame propagation observed involves a well behaved flame front whose transport is radiatively driven. Theoretical relations between these observations and those for our previously observed stabilized lycopodium-air flames have been developed. Additionally, a strikingly novel mode of flame propagation has been observed and analyzed. These latter flames are called "chattering flames" and result from flame-induced acoustic excitation of the Kundt's Tube phenomenon. This leads to spatial segregation of the fuel particles into regularly arranged laminae. Both adiabatic and nonadiabatic analyses have been carried out. Flame-induced acoustic segregation of particulates implies important safety-related consequences for systems of combustible particulates in reduced gravity as well as in normal gravity.

Publications

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Presentations

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Scientific Support for a Space Shuttle Droplet Burning Experiment

Princeton University
Professor F. L. Dryer
Professor F. A. Williams, UCSD
NAS3-24640 (NASA Contact: John Haggard, LeRC)
November 30, 1987 - November 30, 1988

Objectives: The general objective of this program is to ascertain how best to make use of reduced gravity to pursue scientific investigations of droplet combustion. The specific objective is to provide scientific support during development of a droplet burning experiments that are to be carried out in the NASA LeRC drop towers and in the Space Shuttle. The planned experiments are intended to improve our understanding of droplet combustion, especially in relationship to time-dependent and extinction phenomena.

Research Task Description: The research tasks include theoretical modeling of droplet burning, ground-based experimentation on droplet burning, support to NASA in providing advice on hardware aspects of the flight experiment and analysis of data to be obtained in the experiment. The modelling addresses questions related to burning rates, to soot behavior, to disruption, and to ignition and extinction phenomena. Ground-based experiments are focused on droplet ignition and on impulses imparted to droplets by ignition sparks; spark designs for minimum impulse are addressed. In addition, drop tower experiments are addressing burning rates and mechanisms of soot production and of droplet disruption during combustion. The support activities include advisory participation in planning and in implementation of the flight experiment on droplet combustion.

Progress to Date: A ground-based study of impulses imparted to droplets by ignition sparks has been completed. This research identified pressure-induced forces, shock-induced flow and cold-gas flow ahead of the expanding spark kernel as the major contributors to droplet motions. The droplet impulses were established to be of sufficient magnitudes to necessitate spark balancing for achieving spherically symmetrical combustion.

A number of tests on droplet combustion were completed in the 2.2 second dropl tower at NASA LeRC. The fuels studied were decane, heptane, and methanol, the first one in somedetail and the last two in exploratory drops. The atmospheres employed were mainly normal air at atmospheric pressure, although some tests were made in nitrogen-oxygen mixtures at 50% oxygen mole fractions and at total pressures of 0.5, 1.0, and 2.0 atm. It was established that for decane in air, sooting is strong for stationary droplets, and droplet disruption frequently occurs. Burning histories were found to depend strongly on the fuel type and atmospheric conditions.

Theoretical analyses were developed for describing the histories of buildup of foreign constituents within droplets during combustion. The nature of the boundary layer within the liquid at the droplet surface was characterized to enable conditions for the occurence of disruption to be calculated.

Digital data-processing techniques for improving precision o droplet-diameter measurements were developed which increase the precision of the measurement by an order of magnitude.

Publications

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C. PHYSICS AND CHEMISTRY EXPERIMENTS (PACE)

Bubble-In-Liquid Mass Transport Phenomena in a Reduced Gravity Environment

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Professor Kenneth J. De Witt
C-N. Yung
J. L. Brockwell, Union Carbide Technical Center
NAG3-34
(NASA Contact: Dr. A.T. Chai, LeRC)
November 15, 1983 - January 14, 1987

Objectives: The long term objective of the experiment is to observe the dissolution of an isolated gas bubble which is initially of specified size and composition in a thermostatted solvent liquid of known concentration in the reduced gravity environment of earth orbit. The critical radius phenomenon is utilized to maintain the bubble in a state of unstable equilibrium. Subsequent bubble dissolution from the stable critical state is initiated by a step increase in the liquid pressure. A numerical model has been developed which simulates the bubble dynamics under reduced gravity conditions and can be used to determine transport parameters by comparison with the experimental data. The results will yield information on mass transport that will have application to many problems in physical chemistry and technology.

Research Task Description: The experiment involves the observation and measurement of dissolution of an isolated gas bubble which is initially of specified size and composition in a thermostatted solvent liquid of known concentration in the reduced gravity environment of earth orbit. Feasibility of bubble injection and deployment has been shown both in normal gravity using mutually buoyant fluids and under reduced gravity conditions in the NASA Lear jet. Use of the critical radius phenomenon for bubble initialization has been demonstrated to be feasible. Subsequent bubble dissolution or growth from the stable critical state is initiated by a step increase or decrease in the liquid pressure. The justification for conducting the experiment in space is the elimination of large-scale buoyant bubble motion and free convective mass transfer effects that occur in normal gravity. These effects generally mask the molecular mass transfer and interfacial contributions to the dissolution process, and their elimination would allow a more fundamental understanding of the molecular mass transfer and interfacial effects to be obtained.

Progress to Date: Work completed during the current period of performance involved experimentation in two areas: measurement of parameters governing the critical radius phenomenon and demonstration of bubble generation and deployment in microgravity. The critical radius phenomenon has been verified in normal gravity for the CO_2-toluene system in a prototype experiment package constructed at NASA Lewis. Two injectors have been used to satisfy bubble generation and deployment. The first injector relies on gas inertia for deployment, and has been successfully tested on the Learjet. The second injector uses has inertia to generate the bubble and an additional liquid push to deploy the bubble. This injector has been successfully tested using mutually buoyant fluids in normal gravity. The Science Requirements Document has been completed and the Conceptual Design Review is imminent.

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Publications

Yung, C.-N., De Witt, K. J., Brockwell, J. L., McQuillen, J. B., and Chai, A-T., "Factors Affecting the Rate of Bubble Dissolution," J. Colloid & Interface Sci., 1988 (accepted).

Presentations

De Witt, K. J., Brockwell, J. L., Yung, C-N., Chai, A-T., McQuillen, J. B., Sotos, R., and Neumann, E., "Mass Transport Phenomena Between Bubbles and Dissolved Gases in Liquids under Reduced Gravity Conditions," *26th AIAA Meeting*, Reno, Nevada, January 1999, Paper No. AIAA 88-0450.

Determination of the Correlation Length in Helium II in a Microgravity Environment

University of Oregon
Dr. Russell J. Donnelly
Dr. Charles E. Swanson
Benjamin You
Dennis N. Kessler
Daniel T. Smithey
(NASA Contact: D. D. Elleman, JPL)

Objectives: The objective of this research is to measure finite size effects in thermodynamic response functions near a critical point (the lambda transition, T_λ , in liquid helium). Finite size effects are manifested as a rounding ofthe divergence in a thermodynamic function as the correlation length becomes o the order of the system size, so finite size effects occur in a range determined by the correlation length. We can thus test renormalization group group theory predictions and universality assumptions.

In particular, we intend to measure the isobaric expansion coefficient of liquid helium confined between parallel plates. The eventual measurements will cover a range of temperatures very near T_λ (both above and below), a range of pressures, and a range of parallel plate separation distances. In order to measure finite size effects very near T_λ, we need a plate of separation which would result in an extremely inhomogeneous sample in the earth's gravitational field; a microgravity environment eliminates this problem.

Research Task Description: We will determine the isobaric expansion coefficient of liquid helium confined between parallel plates by measurement of the density of hrlium as a function of temperature. We will calculate the density from measurements of the dielectric constant of liquid helium near T_λ . The measurement method involves observing the change in resonant frequency of a reentrant superconducting L-C cavity with liquid helium contained between the plates of the capacitor. Our current apparatus, loaned to us by NBS and NASA Langley whoc designed and built it but never successfully operated it, is suitable for work at only one pressure (essentially SVP). Before flight, we intend to have the ability to work at the full range of liquid pressures.

The apparatus has 3 superconducting capacitors in a single superconducting niobium structure. Two cavities are for the primary measurement, one with a 5μ gap in which to see finite size effects and the other with a 50μ gap for a control. The third cavity is a gas thermometer. The experiment itself will consist of determining the resonant frequencies of the three cavities at various temperatures (indicated by the gas thermometer cavity).

The immediate task is to make the measurements, that is by counting the frequency of the cavity as part of an active oscillator with a tunnel diode as an amplifier. A further task will be to evaluate other methods of determining the resonant frequency, such as coupling a low noise amplifier to the circuit, or measuring the phase change of a signal transmitted through the cavity by a double balanced mixer (probably at low temperature).

Progress to Date: We have been resurrecting the NBS apparatus. We have installed thermometers, a level indicator, relief valves and done some necessary rewiring. We have started to operate the apparatus and found it plagued by leaks. Most of these have

occurred around old indium O-rings, which have now been replaced (necessitating a large amount of rewiring, which is now complete).

The heart of the apparatus, the niobium cavities, appear to be in good condition. All of them have reasonable resonances at room temperature. The temperature cavity has been tested at helium temperatures and has a Q of 10^5, good enough for at least the preliminary work that we are now engaged in. Tests of the other cavities at helium temperatures will be completed shortly.

We encountered a substantial delay when the spectrum analyzer suppled this winter by HP on a ggrant turned out ot be the wrong model and has to be returned. We were able to continue with a spectrum analyzer borrowed from JPL, but it is not nearly as well suited to our requirements and takes much longer for the same measurements. Re-delivery from HP should occur this fall.

Cryogenic Equivalence Principle Experiment

W. W. Hansen Laboratories of Physics
Dr. C.W.F. Everitt
Dr. Paul W. Worden

Objectives: The objective of this research is to test the equivalence of inertial and passive gravitational mass in an earth-orbiting satellite. Preliminary work and technology development is being done in a ground-based experiment which is expected to test the equivalence principle to a few parts in 10^{13}; a satellite version might have a sensitivity of one part in 10^{17}. The apparatus may also be used in a search for composition-dependent short-range forces.

Research Task Description: The ground-based experiment is now well developed. It consists of comparing the motions of two cylindrical test masses suspended in precision superconducting magnetic bearings and free to move along the horizontal (axial) direction. The masses are now made of niobium and lead-plated aluminum but may also be made from any material coated with superconductor. A position detector based on a SQUID magnetometer measures the differential motion between the masses. The periods of the masses are matched by adjustment of the position detector until the system is insensitive to common mode signals, so that the experiment is less sensitive to seismic vibration. The apparatus is contained in a twelve inch helium dewar suspended in a vibration isolation stand. The stand achieves 30 db isolation from horizontal motions between 0.1 and 60 Hz, by simulating the motion of a 200 meter long pendulum with an air bearing. With this attenuation of seismic noise and a common mode rejection ratio of 10^5 in the differential mode, the ground based apparatus should have a sensitivity to equivalence principle violations of one part in 10^{13}. The primary limitation is due to seismic noise.

The earth-based apparatus will be appropriately scaled and modified for operation in zero gravity. The test masses will be about 10 centimeters in diameter. A crucial difference in the orbital experiment is the effect of the gravity gradient of the earth on the masses. This can be eliminated by putting the centers of mass of the test bodies at the same location. If the centers of mass are not coincident, the resulting acceleration can be detected and used as a error signal for a servo loop to drive them into coincidence. The Shuttle version of the experiment should have a sensitivity of about 10^{-15} limited by the vibration environment and gravity gradient field of the Shuttle orbiter. An independent drag-free satellite is necessary for the ultimate version of the experiment which might exceed a sensitivity of 10^{-17} limited by gas pressure effects.

Publications

Worden, P. W., "Almost Exactly Zero: The Equivalence Principle," Near Zero, 1986 (in press).

Worden, P. W., "Measurement and Control of Disturbing Forces on an Equivalence Principle Experiment," in *Proceedings of International Symposium on Gravitational Physics*, China, 1987 (in press).

Zeno: Critical Fluid Light Scattering

Professor R. W. Gammon, University of Maryland
Dr. J. N. Shaumeyer, University of Maryland
Dr. M. R. Moldover, NIST
NAG3-727, May 15, 1986 - December 15, 1987
NAG3-849, December 15, 1987 - March 15, 1989
(NASA Contact: Dr. R. Lauver, LeRC)

Objectives: The objective is measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments are severely limited on earth by the presence of gravity which causes large density gradients in the sample. The goal is to measure fluctuation decay rates at least two decades closer to the critical point than is possible on earth, with a resolution of 3 microkelvin. This will require loading the sample to 0.1% of the critical density and taking data as close as 100 microKelvin to the critical temperature (Tc = 289.72 K). The minimum mission time of 100 hours will allow a complete range of temperature points to be covered, limited by the thermal response of the sample. Other technical problems have been addressed such as multiple scattering and the effect of wetting layers.

Research Task Description: We have demonstrated the ability to avoid multiple scattering by using a thin sample (100 microns), and a temperature history which can avoid wetting layers, a fast optical thermostat with satisfactory microcomputer temperature control and measurement, and accurate sample loading. There remain the important engineering tasks of mounting the experiment to maintain alignment duringlight and using vibration isolation to prevent Shuttle motions from distorting the sample.

The experiment entails measurement of the scattering intensity fluctuation decay rate at two angles for each temperature and simultaneously recording the scattering intensities and sample turbidity (from the transmission). The analyzed intensity and turbidity gives the correlation length at each temperature and locates the critical temperature.

The fluctuation decay rate data set from these measurements will provide a severe test of the generalized hydrodynamics theories of transport coefficients in the critical region. When compared to equivalent data from binary liquid critical mixtures they will test the universality of critical dynamics.

Progress to Date: The current lab version of the experiment has been used to demonstrate the necessary alignment techniques, automated operation of the experiment including a high resolution search for T_c, multiple correlation collection to improve accuracy of decay rate measurements, long term digital integration for high precision transmission measurements. A series of new optical cells with 100 micron optical path, accurately filled with Xe have been produced and studied with regard to sample purity and the time to form gravitational density stratification. The sensitivity of the correlation light scattering apparatus to vibrations has been studied for the purpose of setting requirements for vibration isolation during flight.

Publications

Law, B. M., Gammon, R. W., and Sengers, J. V., "Light-Scattering Observations of Long-Range Correlations in a Nonequilibrium Liquid," Phys. Rev. Lett. 60, 1554 (1988).

Shaumeyer, J. N. and Gammon, R. W., "Optimizing Fitting Statistics in Photon Correlation Spectroscopy," in *OSA Proceedings on Photon Correlations Techniques and Applications*, 1988 (in press).

Presentations

Gammon, R. W., "Photon Correlation Light Scattering Apparatus for the Space Shuttle", presented at the Society of Photo-Optical Instrumentation Engineers (SPIE), August, 1987.

Gammon, R. W., "Description of the Photon-Correlation Instrument for Zeno", NASA Laser Light Scattering Advanced Technology Development Workshop, NASA-LeRC, September, 1988.

Gammon, R. W., "G-jitter: Measurement and Influence on Apparatus", NASA Vibration Isolation Technology for Microgravity Science Advanced Technology Development Workshop, NASA-LeRC, September, 1988.

Briggs, M. E., Shaumeyer, J. N., and Gammon, R. W., "Automated Location of the Critical Point of Fluids and Liquid Mixtures Using Turbidity," Bull. Am. Phys. Soc. 33, 583 (1988), presented at the March APS meeting.

Boukari, H. and Gammon, R. W., "New Expressions to Fit the Damped Harmonic Oscillator Spectrum in a Multi-Pass Fabry-Perot Spectrometer", Bull. Am. Phys. Soc. 33, 735 (1988), presented at the March APS meeting.

Experimental Investigation of Surface Tension Driven Convection as a Feasibility Study for a Microgravity Experiment

University of Texas
Professor E. L. Koschmieder
NAG3-393
(NASA Contract: J.A. Salzman, LeRC)
July 1988 - July 1989

Objectives: The objectives of the planned set of experiments is to establish an experimental data base in order to assess the feasibility and outcome of surface tension driven convection experiments in microgravity. In particular we want to establish the critical temperature gradient required for the onset of convection and the form of the pattern of convection. The second objective of these experiments is to provide sufficient information to compare the experimental results with the theory of surface tension driven convection by Pearson, which theory assumes zero gravity.

Research Task Description: This effort is the experimental investigation of surface-tension-driven convection under supercritical conditions. While the previous studies were concerned with pattern formation under just critical conditions, which are determined by Pearson's theory, we will now investigate the flow at temperature differences larger than the critical temperature difference. We do this following a recommendation of the PACE Science Review Board. If a surface tension driven convection experiment is made in microgravity then one should use this unique opportunity and extend the experiment into the supercritical range, although Pearson's linear theory is not applicable and no theory exists as yet to predict the outcome of such experiments. From our experience with buoyancy driven Rayleigh Benard convection it appears that it will not be easy to develop a nonlinear theory of surface tension dirven convection. The experimental results of supercritical surface tension driven convection in microgravity will therefore be a most useful guide for future theoretical investigations.

The second task of the current set of experiments is the investigation of time requirements for convection experiments in microgravity. This is a matter of practical concern. The time required for a convection experiment to be in equilibrium is the so-called relaxation time, of which there are two, the vertical and the horizontal. While the vertical relaxation time can be easily accommodated on the shuttle, the horizontal relaxation time can be quite long in containers of a couple of inches diameter. The purpose of the planned investigations is to find ways to reduce the horizontal relaxation time to practical time-spans.

Progress to Date: After a two year iterruption we have put our apparatus into operation again and completed the first series of supercritical convection experiments. We have observed the formation of almost perfect patterns of hexagonal convectional cells in a two millmeter deep layer of silicone oil of 100 cs viscosity in a circular container of 4 inches diameter. We have increased the applied vertical temperature difference slowly, and found to our surprise a clear increase of the number of the hexagonal cells. This means that we have observed a decrease of the wavelength of the surface tension driven convective motions. This behavior is just opposite to the well established increase of the supercritical wavelength in buoyancy driven Rayleigh Benard convection. After increasing the temperature to about 1.5 times the critical temperature difference we have decreased the temperature difference again. There may be a nonuniqueness of the supercritical wavelength, but we do not have sufficient data yet in order to make a

definite statement. We have ordered a new reflector of optical quality on a well conducting substrate in order to replace the glass mirror which we have used so far for the shadowgraph visualization of theflow. Having solved the procedural problems for thesupercritical convection experiment, we are set for the final runs as soon as the new mirror arrives.

Lambda Point Experiment

Stanford University
Dr. John A. Lipa
Dr. William M. Fairbank
JPL 957448

Objectives: The objective of the research is to perform a new test oı the theory of cooperative or second order phase transitions by making use o the microgravity conditions on the Shuttle, the JPL SL-2 Helium Dewar, and the Stanford High Resolution Thermometer.

Central to the study of cooperative transitions is the idea of asymptotic behavior of various thermodynamic properties in the limit as the temperature interval from a transition is reduced to zero. Most current theoretical predictions are made in this limit. The present experiment is designed to explore the submicrodegree region of a cooperative transition using new thermometry technology which pushes the resolution of temperature close to the fundamental limits set by statistical fluctuations, near 10^{-12} deg.

The experiment plan calls for the observation of the temperature dependence of the heat capacity of helium very near the lambda point at 2.17K with a resolution of a few times 10^{-10} deg and an accuracy of 1%. This experiment is being conducted in conjunction with JPL, who provides the cryoenic facility. By performing the measurements in space the resolution of the experiment can be improved by a factor of about 100 due to the reduction of the hydrostatic pressure head relative to the situation on the ground. The measurements will lead to a much stronger confrontation between theory and experiment than is possible on earth, and perhaps as severe a test as is feasible with current technology. If the experiment does cast doubt on the theory, it could also have a significant impact on other areas of physics. For example, theories of quark confinement in high energy physics draw heavily on the same type of analysis as used for phase transitions. A major revision of the theory could thus have quite a large ripple effect in theoretical physics.

Research Task Description: The basic objective of the experiment is to measure the form of the heat capacity function in the region very close to the transition. The most important parameter characterizing this function is the exponent which determines the strength of the singularity at the transition. This is derived from the heat capacity data as a function of temperature by a non-linear least squares curve fitting technique. The basic data is obtained by applying precise heat pulses to the sample of helium and measuring the resulting temperature increments with the high resolution thermometers. To obtain results of the desired accuracy it is necessary to control the thermal environment of the sample to high precision. Ths the instrument primarily consists of a multi-stage cryogenic thermostat system with inner control to within $\pm 10^{-8}$ deg. This assembly is mounted within the superfluid helium dewar operating at about 1.7K. Surrounding the dewar is a magnetic shield to reduce the effects of the earth's field on the thermometers. An electronics control system is designed to perform a complete calibration and data collection sequence in an autonomous mode, with ground intervention possible at any time. Since the value of the results increases with the accuracy and resolution of the measurements, the experiment is designed to run continuously throughout the Shuttle flight. The experiment is sensitive to low levels of acceleration, so the maximum possible duration of quiet times will be sought. In addition, a SAMS system will be used to collect acceleration data whenever high

resolution measurements are in progress.

Progress to Date: The main activity in 1988 has been the fabrication and testing of the flight electronics and cryogenic instrument. As of writing, the electronics fabrication has been completed and the instrument piece parts are being machined. Flight software has been developed to the point where it fully supports electronics testing. The high resolution thermometers have been completed and meet all the requirements for the mission.

Publications

Marek, D. and Lipa, J. A., "High Resolution Second Sound Velocity Measurements near the Lambda Point of Helium," Phys. Rev. B 38, 4465 (1988).

Lipa, J. A., Li, Q., Chui, T.C.P., and Marek, D., "Testing the Renormalization Group Theory of Cooperative Transitons at the Lambda Point of Helium," in *Proceedings of the 3rd University of California Conference on Statistical Mechanics*, 1988, Nuclear Physics B (in press).

Presentations

Lipa, J. A., "High Resolution Tests of the Renormalization Group Theory near the Lambda Point," Invited Talk, American Physical Society, New Orleans, March 1988.

Critical Transport Properties in Liquid Helium under Low Gravity

Duke University
Dr. Horst Meyer
Dr. Robert Behringer
NAG5-379 (NASA Contact: Stephen Castles, GSFC)
January 1, 1988 - October 1, 1988

Objectives: The objective of this research task is to measure the shear viscosity η in helium near the liquid-vapor critical point T_c and near the normal-superfluid transition $T_\lambda(x)$ in particular in ^3He-^4He mixtures near the critical point ($X_t = 0.67$, $T_t = 0.87K$) where X and T are the ^3He concentration and T temperature. As the critical point is approached, η diverges, but gravity produces a rounding of this divergence. In the absence of gravity, this rounding will be suppressed and the predicted divergence should be observed until frequency effects (from the measuring oscillator) should be observed that give another rounding closer to T_c. Near the tricritical point in mixtures, a similar behavior can be expected but so far there are no high quality measurements, and our program is to conduct a systematic study of η near T_t. For this purpose, the viscosity of mixtures near $T_\lambda(x)$ (that terminates at the tricritical point) needs to be studied in mixtures with various concentrations $O<X<X_t$. No such systematic study has ever been made, and it is necessary to understand the trend of the singular behavior, crossing over from the "critical" to the "tricritical" regime. We are in the process of doing this and we are moving systematically towards measurements at lower temperatures.

When η measurements near T_t are completed, we will be able to decide whether such measurements are more interesting than those near the liquid-vapor transition, for a shuttle space slight or on a space platform. And most important, are they <u>feasible</u> given the experimental constraints and requirements (long equilibrium times, temperature stability needs, etc.)? Are the experiments really <u>good candidates</u> for a flight under low gravity conditions and justify the considerable expense in money and time?

Research Task Description: The viscosity is measured by means of a torsional oscillator operating in a continuous mode at a freqency of 150 Hz. The quality factor Q_T of the oscillator-a flat thin circular cell containing a space for a fluid layer 0.04 cm high and 5 cm diameter connected to a base via a torosional rod- is measured by the ratio of the detected AC voltage (proportional to the oscillator amplitude) and the driving voltage. Measurement of the empty oscillator performance gives Q_E^{-1}. Finally one obtains the damping from the fluid, $Q_F^{-1} = Q_T^{-1} - Q_E^{-1}$, where Q_F^{-1} is proportional to $(\rho\eta)^{1/2}$ and ρ is the mass dendity [In the superfluid phase $Q_F^{-1} \propto (\rho_n\eta_n)^{1/2}$, where n denotes the normal component, is measured]. We use the arrangement described in ref. 1 for the oscillator and electronic control. The temperature of the cel is controlled to about $1\mu K$, and the superfluid transition is defined by the sharp change in the slope $d(\rho\eta)/dT$ at T_λ.

The precision for our first oscillator was 2×10^{-4}. However, the performance inexplicably deteriorated, and a new oscillator was built, having a precision of 6×10^{-4}. To obtain η from the measured product $\rho\eta$, the density ρ has to be measured separately, a task we are preapring for presently. We use a cell where the dielectric constant of the fluid is measured. The density is then obtained via the Clausius Mossotti relation.

Progress to Date: In the past ten months, we have measured with our torsional oscillator the product ($\rho\eta$) on ixtures with X = 0.00, 0.03, 0.3, 0.4, 0.5 and 0.65. We were

slowed down by long equilibration times, and by the many data points to be taken close to $T_\lambda(X)$, which is very time consuming. A considerable amount of time was spent finding and repairing some electronic problems in our data taking system and in developing new software for the recently acquired MacII computer superseding an old microprocessor which tended to break down.

We have completed the construction of a torsional oscillator of a different design for further measurements of ρ in the tricritical region. This oscillator is undergoing test at present.

Also we are preparing to test a density cell to measure ρ in mixtures between 1.2 and 4 K. Combination of the ($\rho\eta$) data with those of ρ will give $\eta(T)$ for the various mixtures.

Publications

Meyer, H., "The Transport Properties of ^{3}He-^{4}He Mixtures near the Lambda Line--A short Review and Outlook," J. Low Temp. Phys. 70, 219 (1988).

Meyers, H., "Methods for the Measurement of Transport Properties of Fluids at Low Temperatures," in preparation.

Wang, S., Howald, C., and Meyer, H., "Shear Viscosity of ^{3}He-^{4}He Mixtures near the Superfluid Transition," in preparation.

Precise Viscosity Measurements Very Close to Critical Points

Thermophysics Division, National Institute of Standards and Technology
Dr. M.R. Moldover
Dr. R.F. Berg
Professor R.W. Gammon, University of Maryland
C-32001-K NASA Contact: Dr. R.A. Wilkinson, LeRC
January 1, 1988 - August 31, 1988

Objectives: Our objective is to accurately measure the viscosity of a pure fluid near its liquid-vapor critical point. The space experiment will be the fourth of a series of tasks which are: (1) theoretical studies, (2) critical viscosity measurements of binary liquid mixtures, (3) critical viscosity measurements of pure fluids in 1-g, and (4) measurements of pure fluids in low gravity. We have developed a torsion oscillator viscometer and used it to study four binary liquid mixtures near their consolute points.

Research Task Description: Near the critical temperature Tc the viscosity η diverges as:

$$\eta \sim (T-T_c)^{-y}$$

Analyses of all four binary liquid data sets show that the viscosity exponent is $0.0404 < y < 0.0444$, significantly higher than the theoretically predicted value of 0.032. These results are described in the last reference below.

The low frequency, low shear viscometer used for the binary liquid measurements has been redesigned to measure pure fluids. The new thermostat is smaller and fully automated and the new sample cell is a disc-shaped container of 1 mm height and 15 mm radius. These dimensions were chosen to minimize the undesirable effects caused by shear, gravity, thermal diffusivity, and loading inaccuracy near the critical point.

Progress to Date: We have measured the viscosity of CO_2 over a 5 decade range of reduced temperatures. The effect of gravity on the critical divergence was clearly important within 10 mK of T_c. Also, near T_c very slow thermal relaxation caused noticeable hysteresis unless the sweep rate was less than about 0.2 microkelvin per second. Preliminary analysis indicates the experimental viscosity exponent to be in better agreement with theory than for the binary liquids.

We are also numerically studying theoretical models of the slow thermal relaxation near T_c. Understanding this effect is crucial for successful low gravity liquid-vapor critical point measurements

Publications

Berg, R. F. and Moldover, M. R., "Critical Exponent for the Viscosity of Four Binary Liquids", J. Chem. Phys. 89, 3694 (1988).

Studies in Electrohydrodynamics

Princeton University
Professor Dudley A. Saville
NAG3-259
(NASA Contact: Dr. R. Balasubramanian, LeRC)
February 1982 - April 1989

Objectives: The purpose of this work is to develop and test models of electrodynamic processes involving fluid interfaces. Particular attention is given to the behavior of fluid globules in systems with poorly ionized solutes at high (applied) field strengths. An ad hoc model (the leaky dielectric) described by G.I. Taylor and J.R. Melcher will be expanded so as to include electrokinetic effects and put it on a form theoretical basis.

Research Task Description: Extant theories which account for the details of the physico-chemical processes associated with charged interfaces deal exclusively with low field strengths and fully ionized solutes. The model now used to describe processes at high field strengths -- the leaky dielectric -- omits consideration of electric double layers, adsorption at interfaces, and chemical processes involved in the dissociation and recombination of solute species. Thus, even though the model depicts some features associated with bulk fluid motion faithfully, it fails to give a comprehensive picture.

The research involves several tasks: (1) construction of a mathematical model for low field strength electrokinetics with fluid globules wherein the interface is permeable to ions and the electrolyte contains poorly ionized solutes, (2) extension of the model described in (1) to high field strengths, (3) adaption ofthe model to oscillating fields and (4) experiments involving oscillating fields with fluid globules to test the theory. The experiments will take advantage of a microgravity environment to overcome problems associated with sedimentation and buoyancy driven convection arising from Joule heating.

Progress to Date: The theoretical work is now largely complete for steady fields. We first first studied the low field strength electrophoresis of drops with interfaces permeable to ions to gain insight into the more difficult problem of high field strength electrophoresis. The mathematical model devloped accounts for: (1) weak electrolytes, (2) charge regulation at the interface, (3) interfacial tension gradients at the interface, (4) surface transport of ions within the interface, and (5) the permeability of the interface to ions.

The second theoretical problem we attacked concerned the incpetion of field dependent mobility, i.e., the point at which the mobility is no longer proportional to the field strength. This was solved using singular prturbation methods.

The central problem of this research is to describe the high field strength behavior of fluid globules. Using the knowledge gained in solving the problems described earlier, we constructed an asymptotic solution to describe the behavior of an uncharged or charged drop in a strong electric field when the drop is large compared to the Debye thickness. Here the flow and stress fields near the interface differ from those computed according to the leaky dielectric model which ignores electrokinetic effects.

The sample cell and high voltage electronics have been designed, constructed, and assembled for experiments involving a drop (free or pendent) in a liquid. Output from a high voltage (DC) power supply is converted to a sine wave and fed to the two

electrodes. We can vary the frequency up to 100 Hz; voltage across the electrodes ranges from 0 to 15 kV. A CCD camera (Ikegami) is used to image the drop. Our system has been tested with both pendent and free drops. Although many tasks remain to be completed we believe we have the essential elements of a superb system.

Publications

Saville, D. A. and Baygents, J. C., "The Migration of Charged Drops and Bubbles in Electrolyte Gradients," Physicochem. Hydrodynam., 1988 (in press).

Presentations

Saville, D. A. and Baygents, J. C., "The Circulation Produced in a Drop by an Electrostatic Field: A High Field Strength Model," Third International Colloquium on Drops and Bubbles, Monterey, CA, September 1988.

Saville, D. A. and Baygents, J. C., "Electrohydrodynamics: The Circulation Produced in a Drop by an External Electric Field," Division of Fluid Dynamics Meeting, American Physical Society, Buffalo, November 1988.

Saville, D. A. and Baygents, J. C., "The Migration of Small Particles in Electrostatic Fields: Nonlinear Electrophoresis," AIChE Annual Meeting, Washington, DC, November 1988.

Mechanics of Granular Materials

University of Colorado, Boulder
Dr. Stein Sture
NAS8-35668
(NASA Contact: I. C. Yates, MSFC)
November 2, 1983 - July 31, 1988

Objectives: This research effort is aimed at understanding the constitutive behavior of granular materials subjected to very low intergranular stress levels. Specifically the goal is to characterize stress-strain response, strength behavior, and dilatancy properties of dense and mdeium dense granular assemblies at low effective confining stresses.

Research Task Description: Analytical constitutive models for granular materials that can predict stress-strain response, strength, and dilatancy at very low as well as high mean stress levels have been devised. The most challenginf aspect of this work has been the characterization of response when very dense granular packings are sheared at very low stress levels. The dilatancy rate is the very highest in this case, and its is difficult to devise functions that do not violate solid and continuum mechanics concepts.

Progress to Date: Techniques for integrating the constitutive equations in nonlinear finite element analysis computer codes have been developed. A series of ground-based experiments have been conducted, where the self-weight of the specimen resulted in unhomogeneous deformation and stress fields. The specimens have been analyzed as full-fledged boundary value problems and material properties have been assessed via inverse identification procedures.

Publications

Sture, S., Runesson, K. R., and Macari-Pasqualino, E. J., "A Three-Invariant Plasticity Model for Granular Materials," J. Appl. Mech., 1988 (in press).

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D. FACILITIES

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Microgravity Materials Science Laboratory

NASA Lewis Research Laboratory
Thomas K Glasgow
In-House
February 1984--continuing task

The Microgravity Materials Science Laboratory (MMSL) was created to serve as a focal point for ground-based experimentation in preparation for or conjunction with flight experiments. It is open to users from industry, academia, and government. The MMSL addresses a broad range of materials including metals, alloys, salts, glasses, ceramics, and polymers. The laboratory is co-supported by the Office of Commercial Programs and the Office of Space Science and Applications.

The laboratory is equipped with a wide variety of apparatus for characterizing the interaction of liquids or gasses with gravity during materials processing. Included are transparent furnaces for observation of salt solidification or physical vapor transport, an electromagnetic levitator for containerless melting of metals, a bulk undercooling furnace, a magnetically damped directional solidification furnace, a transparent isothermal dendrite growth apparatus, a transparent model directional solidification furnace, a hot stage (1600C) optical microscope, a functional duplicate high temperature acoustic levitator, glass melting and characterization equipment, polymers preparation and characterization equipment, a metallography laboratory, and computational facilities for process modelling. Most of the equipment operates under computer control. New equipment is acquired or built in response to specific requests. The staff includes engineers and technicians drawn from diverse disciplines including mechanical, nuclear, metallurgical, and welding engineering, physics, and chemistry.

In the past year the laboratory facilities have been used for a variety of projects and experiments including directional solidification of alloys, polymer composite experiments, glass refining, glass quenching, electro-optic crystal growth, telescience trials, characterization of KC-135 samples, thermal energy storage work, characterization of polymers, and bulk undercooling. The computational modelling facilities have been especially busy this year with examination of proposed flight experiments. The research performed is reported under the respective tasks.

The MMSL is also home to two Advanced Technology Demonstration projects, Laser Light Scattering and High Temperature Furnaces. Potential users of the MMSL should request the brochure "The Microgravity Materials Science Laboratory" for a more complete description and application for use.

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Ground-Base Research Facilities

NASA Lewis Research Center
Jack Lekan
In-House
January 1, 1988- October 31, 1988

The Space Transportation System and eventually the Space Station offer opportunities for long duration (minutes,hours, days, weeks) scientific research and technology development under microgravity conditions. The resources that are required to utilize such facilities demand thorough scientific justification and technological feasibility determinations before committing to a space-based experiment. Several NASA ground-based facilities at the Lewis Research Center (LeRC) are employed to obtain the baseline, normal gravity data and preliminary reduced gravity data (gravitational levels of 10^{-2} to 10^{-6} g) that are necessary to advance the scientific understanding and experiment concepts. The facilities that can provide the prerequisite low-gravity environment include the 2.2 Drop Tower, 5.18 Second Zero-Gravity Facility and a model 25 Learjet which provides up to 20 seconds of experimental test time.

The recent two and one half year period without Space Shuttle flights, coupled with potential longer range limitation on available Shuttle manifest opportunities for experiments, increased the interest in expansion of the microgravity scientific data return from ground-based low-gravity facilities. Thus, while space experiment hardware technology development and science requirements definition were continuing to be major activities at the facilities, many experiment programs were re-examined to determine how additional ground-based reduced gravity data might be utilized to enhance the success and value of limited space-based experiment time. Expanded ground-based test programs were defined and initiated for several experiment programs to obtain unique microgravity data which could be used to refine current analytical models of the processes and phenomena under investigation. These data and improved models may either stand-alone without the need for additional flight data or may be used to refine the final test matrices and experimental techniques that will be used in future space-based experiments. A brief description of the LeRc facilities and summary of the 1988 highlights for each facility follows.

The 2.2 Second Drop Tower is extensively utilized by NASA research scientists and experiment designers as well as principal investigators for the academic community who are conducting research in the areas of Combustion and Fluid Dynamics and Transport Phenomena. As the name implies, this facility provides 2.2 seconds of low gravity test time for experiment packages up to 125 kilograms of hardware weight. An experiment is enclosed in, but not attached to a drag shield. During a test drop, the experiment package and drag shield drop freely and independently from each other a distance of 27 meters. The drop shield reduces aerodynamic drag on the experiment during the fall. Gravitational accelerations experienced by the experiment are 10^{-5} g. The rapid experiment turnaround time (8 to 12 tests can be performed per day) and relative low cost of operation, make this facility particularly attractive to a variety of investigators.

The 2.2 Second Drop Tower celebrated its most ambitious year in 1988 as a total of fifteen programs were supported. Five new experimental packages were designed, fabricated, assembled and implemented. The inventory of active experimental packages has grown from four to twelve over the last year and one -half. Over 650 research drops were performed. Also during this time period, the facility executed its 8000th

research drop.

The following experiments were supported by the 2.2 Second Drop Tower in 1988: Burke-Shuman Diffusion Flames, Cellular Flames, Droplet Combustion, Forced Convective Flame Spread, Fuel Droplet Vaporization, Gas Diffusion Flames, Opposed Flow Flame Extinction, Particle Cloud Combustion (ground-based feasibility and flight), Pool Fires: Flame Spread, Pool Fires: Pre-ignition, Pre-Mixed Gas Combustion, Smoldering Combustion, Surface Tension Driven Convection and Two-Phase Flow.

A Preliminary Engineering Report for a 1990 construction project for the rehabilitation and modification of the Drop Tower was also completed. Also, the Statement Of Work for the Final Architectural-Engineering Design Study was subsequently prepared. The major components of this project are an 850 square floor addition to the existing facility for shop and laboratory work., an erosion abatement system, and the upgrading of the existing mechanical and electrical systems.

The 5.18 Zero-Gravity Facility is a sophisticated research facility which contains a 145 meter shaft with an integral vacuum drop chamber which can be evacuated to a weight of up to 450 kilograms contained in an experiment vehicle experience gravitational accelerations of 10^{-6} g for 5.18 seconds during free fall in this facility. Other features, such as a telemetry system and closed-circuit television system represent a significant expansion in experiment sophistication and research capabilities, when compared to the Drop Tower. Due to the complexity of facility operations, especially the evacuation of the vacuum chamber, only one test is generally performed per day.

The Zero-Gravity Facility, like the Drop Tower, also experienced extensive utilization in 1988. Six major programs were supported as 135 drops were performed as several of the experiments were built up or underwent extensive modification. The inventory of drop vehicles grew from three to five as dedicated vehicles were provided for the build-up and testing of the Droplet Combustion experiment and initiation of build-up for the Gas Diffusion Flame Experiment. Due to space limitations in the building, the Zero-Gravity Facility is operating at near full utilization.

During 1988, the following experiments were supported: Droplet Combustion, Surface Tension Driven Convection (heater insertion, alternate filling, and inflow tests), Contact Angle Measurements, Pool Fires: Flame Spread (filling and igniter tests), Solid Surface Combustion, and Gas Diffusion Flame.

A specially modified Learjet Model 25, that has the capability of flying parabolic (Keplerian) trajectories, is also utilized for low gravity research. This facility can provide a significant increase in the amount test-time (20 seconds) when compared to the drop towers. However, the gravitational levels of only as low as 10^{-2} g can be attained as experiments are fixed to the interior of the aircraft. This aircraft does provide an additional capability as a researcher can fly along with an experiment to not only monitor it, but also reconfigure the experiment between low gravity maneuvers.

Four experiments were supported by the Learjet in 1988 as 43 flights and 75 trajectories were executed, The support of these experiments included the design fabrication, build-up and testing of new hardware foe the Particle Cloud Combustion Experiment, Vibration Isolation Experiment, and Gas Diffusion Flame Accelerometer System, which will be utilized on the JSC KC-135. Modifications were also implemented for a series of tests with the Two-Phase Flow Experiment package.

E. CENTERS OF EXCELLENCE

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Center for Separation Science

University of Arizona
Dr. M. Bier
NAGW-693
January 1, 1985 - December 21, 1988

The Center for Separation Science has been organized to serve as a center of excellence for NASA and the U.S. biotechnology industry in matters relating to microgravity science and applications. The Center's primary expertise is in electrophoresis, covering a broad range of its subspecialties, including (1) the preparative techniques of zone electrophoresis, isoelectric focusing and isotachophoresis; (2) the mathematical modelling and computer simulation of electrophoretic transport processes, and (3) the analytical techniques of two-dimensional and capillary electrophoresis.

This expertise has been assembled at little cost to NASA, through the generous contribution, by their manufacturers, of instruments from Sweden, Germany and England, as well as the U.S. Thus, the Center is presently recognized by scientists in industry and academia as a unique resource in electrophoresis. This is attested to by numerous visits from industry and its financial support, the presence of postdoctoral fellows supported by German and Korean science grants, and a graduate student supported by the French pharmaceutical industry.

In addition, scientists from the Center have been asked to collaborat in microgravity research projects by the German and French national space organizations, which will lead directly to space experimentation.

The Center is also exploring other novel techniques which may benefit from operation in microgravity. These include techniques based on cell fusion, supercritical fluids and phase partitioning methods.

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Center for Low-gravity Fluid Mechanics and Transport Phenomena

University of Colorado
Professor D.R. Kassoy
Professor R. L. Sani
NAGW-951 (NASA Contact: R.K. Crouch, HQ)
September 1, 1986 - March 31, 1989

The Center has developed an integrated program of research and education in low-gravity science and technology. Participants in the program, now numbering twenty-two faculty, students and research associates from four different departments, focus on the role of fluid mechanics, heat and mass transfer in materials processing, fluid handling, thermal management and combustion. The research projects are interdisciplinary in character. Investigators interact directly with engineers and scientists in laboratories and industries concerned with specific processes and technologies.

The Center has initiated research activity in the following areas:

(1) *Modeling and Experiments on Fluid Systems with G-jitter*
P.D. Weidman
S. Biringen

Computational methods are used to model g-jitter effects in a thermosiphon heat loop and on buoyancy-driven convection in rectangular cavities and three-dimensional boxes. Ground-based g-jitter experiments are designed to measure effects on convection in fluid-filled boxes as well as those with a free surface.

(2) *Fluid Microstructural Effects of Relevance to Low-gravity Material Processing*
W.R. Krantz

The focus of this research is to determine the proper mathematical formulation of dynamic fluid microstructural phenomena and to apply such a formulation to the modeling of the solidification of miscibility-gap composites.

(3) *Low-gravity Effects on Thermomechanically-Induced Convection in Confined Gases*
D.R. Kassoy

Modeling methods are used to ascertain the magnitude of thermoacoustic convection induced in confined gaseous helium by heat transfer from the confining boundary. The competition between thermoacoustic and buoyancy-induced convection at reduced gravity levels is analyzed.

(4) *Modeling of Microgravity Processing of Bimetallic Composite Materials*
R.H. Davis

Modeling of droplet collisions and growth in liquid phase miscibility gap systems under low-gravity conditions is the focus of this research. The interaction between two neighboring droplets of the dispersed phase, as well as the evolution of the drop-size distribution during processing, are being studied.

(5) *Computer-Aided Analysis of the Flow, Transport and Stability of Systems with Free and/or Moving Boundaries 1. Floating Zone Processing*
R.L. Sani

Finite element numerical algorithms are being developed to model the flow, transport and stability of floating-zone processing configurations. Interactions between convection, heat and mass transport, as well as deformable free-surface effects, are emphasized.

(6) *Manufacturing Spherical Shells under Microgravity Conditions*
C.Y. Chow

Electromagnetic-capillary instabilities on liquid metal cylinders are studied in order to determine the feasibility of manufacturing spherical shells from thin-walled metal tubing. Magnetohydrodynamic pinch effects are found to be an effective means for dividing the cylinder into elements of uniform size. Surface tension then generates spherical shells.

(7) *Thermal Instabilities in Low-Prandtl Number Liquids*
J.E. Hart

Experiments in an annular mercury bath heated on one vertical wall and cooled on the other are used to discover an assortment of novel convective instabilities. Finite element as well as finite difference computations are employed to resolve motion not accessible to measurement, and to investigate the mechanism of the instability.

In addition to these University-based research projects another Center participant, H. Snyder, has developed an expertise in fluid-handling problems, particularly superfluid helium transfer through an interaction with Ball Aerospace in Boulder, Colorado.

The Center provides graduate level courses in low-gravity sciences and in materials processing in space. In addition, the University of Colorado provides financial support for the Center-sponsored Science Seminar. This weekly series give both U.S. and international experts an opportunity to report on the latest developments in low-gravity science and technology. Approximately 22 students participate in these educational programs.

The Center fosters extensive interaction with scientists and engineers in other universities, in government laboratories, and in industry. Cooperative research projects are being developed and long-term visits have been arranged. The Center seeks the broadest possible participation by members of both the national and international low-gravity community.

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Institute for Theoretical Physics

University of California, Santa Barbara
J. R. Schrieffer
D. M. Eardley
J. S. Langer

A. Fluid Dynamics

Fluid Dynamics as a broad subfield of physics has seen explosive growth in recent years. Research in this field is being vigorously pursued at the Institute for Theoretical Physics (ITP) in a number of directions including fundamental questions concerning dynamical systems in general, applications to material science, astrophysics and magnetohydrodynamics as well as advanced computational techniques relevant to these fields and to meteorology, oceanography and aerodynamics.

The ITP is structured around six month research programs involving 25-30 participants and focussed on specific topics, in addition, to continuing research activities carried on by the permanent members and post doctorals members. As an example, a research program entitled, *Active Galactic Nuclei*, was held at ITP January - June 1987, with hydrodynamics playing a central role. Several of the key participants and their research focus are as follows:

1. Professor Robert Abramowitz, Trieste; *Hydrodynamics of Thick Accretion Discs*
2. Professor Roger D. Blanford, Caltech; *Magnetic and Plasma Processes Near Black Holes, Winds from Black Holes*
3. Professor Peter Goldreich, Caltech; *Hydrodynamic Instabilities of Thick Accretion Discs; Generation of Hydrodynamic Oscillations by Convection in the Sun*
4. Professor Shoji Kato, Univ. of Kyoto; *Hydrodynamic Instability of Thick Accretion Discs*
5. Professor Arieh Kovigl, Univ. Chicago; *Hydrodynamics and Magnetohydrodynamic Jets at High Mach Number*
6. Dr. Charles Evans, Caltech; *Accretion onto Black Holes; Numerical Hydrodynamics*
7. Dr. John Hooley, Caltech; *Numerical Magnetohydrodynamics*

Numerous papers were published from this program dealing with hydrodynamics.

In addition, a program concerning fundamental problems in statistical mechanics was held September-December 1987, coordinated by Robert Griffiths (Carnegie Mellon Univ.), J. S. Langer (ITP) and Joel Lebowitz (Rutgers Univ.) The relation of hydrodynamics to dynamical systems theory was explored in depth as well as specific problems in nonlinear growth kinetics and pattern formation. This work is closely related to the ongoing research activities of Prof. J.S. Langer and his group at ITP.

A third program, *Computational Fluids Dynamics*, will be held at ITP September-December 1988. This program is being coordinated by Prof. P. Marcus (Berkeley), and S. Teukolsky (Cornell Univ.), with strong input by Prof. D Eardley (ITP). The program will have a balance of "pure" fluid dynamicists and scientists from fields such as astrophysics, general relativity, etc. The program will include hydrodynamics magnetohydrodynamics, plasma physics, and novel numerical techniques. Attention will be given to communicating the most recent concepts and techniques between the disciplines. Some of the key participants in the program, in addition to the coordinators, will be Drs. T. Piran (Hebrew Univ.), R. Wilson (LLL), P. Wirta (Georgia State Univ.), J. Hawley (Univ. VA), S. Finn (Cornell Univ.), D. Marion (Caltech), M. Nauenberg (Santa Cruz), M. Choptuik (Cornell Univ.), W. Press (Harvard), and H. Zalwsky (Univ.). Many of these topics are related to ongoing research of Prof. D. Eardley and his group at ITP. A parallel program on Cosmology and Microphysics coordinated by J. Hartle (Santa Barbara), M. Turner (Univ. Chicago) and F. Wilwzek (ITP) will no doubt interact effectively with this program.

The ITP plans to continue vigorous research in hydrodynamics and related problems in the future.

In addition, the Principal Investigator of the ITP serve on numerous NASA Advisory committees bringing the expertise of the Institute to bear on microgravity science and applications and other divisions. The breadth and cross disciplinary character of the ITP is of particular significance in providing high quality advice for programmatic development in these areas to NASA

B. Kinetics of Dendritic Growth

During the last year or so, Professor Langer's work in solidification theory has focussed primarily on the origin of dendritic sidebranching and the response of dendritic patterns to fluctuations in the temperature and composition of the solidifying material. The results of these investigations have revealed an extreme sensitivity of dendritic structure to minute changes in growth conditions and a serious possibility that sidebranches in many cases of physical interest are actually generated by ambient thermal noise.

New projects in this program have to do with the effects of strong crystalline anisotropy on dendritic growth forms, the transition to interface control that occurs with increasing undercooling or supersaturation, and the stability of dendritic arrays in directional solidification. The latter project is likely to have the most direct implications for microgravity research because it is motivated by the need for a theory of the so-called "mushy zone" that determines solute segregation in the casting of alloys. Current attempts to model thermosolutal convection in the mushy zone must necessarily be based on incomplete fundamental understanding of how this zone is formed and what determines its structure and stability.

The Institute for Theoretical Physics produces a total of 160-180 technical articles per year partially supported by NASA.

APPENDIX A
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16 Abstract

This report is a compilation of the active research tasks as of the fiscal year 1988 of the Microgravity Science and Applications Program, NASA Office of Space Science and Applications, involving several NASA centers and other organizations. The purpose of this document is to provide an overview of the program tasks and to provide the principal investigators, their affiliations, and publications. It is hoped that this publication will be helpful to the program, the discipline, and users in the identification of research areas and in the review, planning, and implementation of the program. The research tasks are described briefly, and a bibliography is provided.

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